

# **EXHIBIT 23**

## **Part 2**

## 9.2 Requirements

**9.2.1 Transmission electron microscope**, operating at an accelerating potential of 80 kV to 120 kV. The TEM shall have an illumination and condenser lens system capable of forming an electron probe smaller than 250 nm in diameter.

**9.2.2 Energy dispersive X-ray analyser**. The TEM shall be equipped with an energy dispersive X-ray analyser capable of achieving a resolution better than 170 eV (FWHM) on the Mn  $K_{\alpha}$  peak. Since the performance of individual combinations of TEM and EDXA equipment is dependent on a number of geometrical factors, the required performance of the combination of the TEM and X-ray analyser is specified in terms of the measured X-ray intensity obtained from a fibre of small diameter, using a known electron beam diameter. Solid-state X-ray detectors are least sensitive in the low-energy region, and so measurement of sodium in crocidolite is the primary performance criterion. The combination of electron microscope and X-ray analyser shall yield, under routine analytical conditions, a peak from sodium that allows discrimination between the spectra from crocidolite and amosite.

**9.2.3 Vacuum coating unit**. If carbon-coated specimen grids are not available, a vacuum coating unit capable of producing a vacuum better than 0,013 Pa shall be used for vacuum deposition of carbon for preparation of carbon-coated grids.

**9.2.4 Calibration grids**. TEM specimen grids prepared from dispersions of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, richterite/winchite, and talc are required for calibration of the EDXA system. It is recommended that gold or nickel grids be used to facilitate detection of sodium. For calibration of the camera constant for interpretation of ED patterns, TEM specimen grids with vacuum-evaporated thin films of gold, aluminium or thallos [TI(I)] chloride deposited on to carbon films are required.

**9.2.5 Disposable tip micropipettes**, suitable for transferring a volume of approximately 3  $\mu$ l to a carbon-coated TEM specimen grid.

## 9.3 Calibration

### 9.3.1 EDXA system

For the purposes of this method, calibration consists of obtaining EDXA spectra from reference samples of chrysotile, amosite, crocidolite, tremolite, actinolite, anthophyllite, and richterite/winchite. The chemical compositions of commercial chrysotile, amosite, crocidolite, and anthophyllite do not vary substantially, and comparison of unknown EDXA spectra with those from the three reference asbestos samples constitutes sufficient identification for this part 1 of ISO 22262. For most purposes, it is not necessary to discriminate between tremolite and actinolite, since the compositional boundary between them is a matter of convention. When it is necessary to discriminate between tremolite and actinolite, the SRM 1867 tremolite and actinolite samples are particularly useful since they have compositions just below and just above the boundary defined by the International Mineralogical Association (Reference [24]). In some applications, the magnesium may be partially leached from chrysotile, leading to a chemical composition that approaches that of talc. In order to facilitate the discrimination between chrysotile and talc or anthophyllite, it is recommended that an EDXA spectrum also be obtained from a known sample of talc. Use this spectrum to define the upper limit of the magnesium mass fraction in talc. Examples of EDXA spectra obtained on the SRM 1866 and SRM 1867 samples, the HSE reference asbestos samples, Bolivian crocidolite, and richterite/winchite appear in Annex F. For positive identification, reference EDXA spectra from asbestos standards similar to those shown in Annex F should be recorded using the specific combination of TEM and EDXA detector, since the geometries and detector efficiencies vary between different instruments.

### 9.3.2 Camera constant for interpretation of ED patterns

Use gold, aluminium or thallos [TI(I)] chloride to calibrate the radius-based camera constant,  $\lambda L$ , the product of the wavelength and camera length, for electron diffraction patterns. Specimen grids with a vacuum deposited, thin, polycrystalline film of one of these materials on a thin carbon film are used for the calibration. The calibration data for the first two diffraction rings, where  $D$  is the ring diameter, are shown in Table 6.

Table 6 — Radius-based camera constants

Calibration material	Radius-based camera constant $\lambda L$	
	1st diffraction ring	2nd diffraction ring
Gold	0,117 74 <i>D</i>	0,101 97 <i>D</i>
Aluminium	0,116 90 <i>D</i>	0,101 24 <i>D</i>
Thallous [Tl(I)] chloride	0,192 14 <i>D</i>	0,135 86 <i>D</i>

## 9.4 Sample preparation

Remove representative fibres from the sample (see 7.2.2 and 7.2.3), and place them in an agate mortar, and pestle. Add approximately 1 ml of ethanol, and grind the fibres with the pestle until they are well dispersed in the ethanol. Set up a laboratory stand and clamp, and use it to hold a pair of fine-point tweezers that are supporting a carbon-coated TEM specimen grid, with the carbon side facing upwards. Using a disposable tip micropipette, drop a 3  $\mu$ l volume of the ethanol dispersion on to the grid, and allow it to dry. Drying is faster if the grid is held under a heat lamp. When dry, the TEM grid is ready for examination.

If crocidolite or sodic-calcic amphibole is suspected, use of a carbon-coated gold TEM grid is recommended in order to avoid partial overlap of the Na  $K_{\alpha}$  peak by the Cu  $L_{\alpha}$  X-ray peak if a copper grid is used.

## 9.5 Qualitative analysis by TEM

### 9.5.1 Acquisition of EDXA spectra

It is important to obtain the EDXA spectrum from clean areas of the fibre, since distortion of peak heights by contributions from attached particles may compromise the identification.

### 9.5.2 Chrysotile

The morphological structure of chrysotile as seen in the TEM is characteristic and, with experience, can be recognized readily. However, a few other minerals have a similar appearance, and morphological observation by itself is inadequate for most samples.

Classify a fibre as chrysotile if:

- the Mg and Si peaks are clear, and comparable in Mg/Si peak height ratio with that of reference chrysotile;
- any Fe, Mn and Al peaks are small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

**IMPORTANT — Anthophyllite and talc both yield EDXA spectra that conform to these specifications, but the Mg/Si peak height ratio for these minerals is lower than that for chrysotile. In order to avoid erroneous classification of talc or anthophyllite as chrysotile, take account of the Mg/Si peak height ratio and calibrate the EDXA detector using known samples of chrysotile and talc.**

### 9.5.3 Amosite

Classify a fibre as amosite if:

- the Mg, Si and Fe peaks are comparable in ratio to those of the reference amosite;
- no statistically significant peaks from Na or Al are present;
- the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

#### 9.5.4 Crocidolite

Classify a fibre as crocidolite if:

- a) the Na, Si and Fe peaks are comparable in ratio with those of the reference crocidolite;
- b) no statistically significant peak from Al is present;
- c) any peak from Mg is small, and no Mn peak is visible.

NOTE 1 Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

NOTE 2 If a large peak from Mg is present, it is possible that the fibre is magnesio-riebeckite. Bolivian crocidolite is the only known commercial source, although this variety of crocidolite can occur as contamination of other minerals.

#### 9.5.5 Tremolite

Classify a fibre as tremolite if:

- a) the Mg, Ca and Fe peaks are comparable in ratio with those of the reference tremolite;
- b) no statistically significant peak from Al is present;
- c) any peak from either Na or K is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

#### 9.5.6 Actinolite

Classify a fibre as actinolite if:

- a) the Mg, Si and Fe peaks are comparable in ratio to those of the reference actinolite;
- b) no statistically significant peaks from Na or Al are present;
- c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

#### 9.5.7 Anthophyllite

Classify a fibre as anthophyllite if:

- a) the fibre is straight and exhibits no evidence of a ribbon-like structure;
- b) the Mg and Si peaks are comparable in ratio to those of reference anthophyllite — anthophyllite from some sources may not exhibit a peak from Fe, although in commercial anthophyllite a peak from Fe will probably be observed;
- c) no statistically significant peaks from Na or Al are present;
- d) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

#### 9.5.8 Sodic–calcic amphibole asbestos (richterite/winchite)

Classify a fibre as sodic–calcic amphibole if:

- a) the spectrum is similar to that of actinolite or tremolite, but the Ca peak is substantially smaller and an Na peak is present — a K peak may also be evident;
- b) no statistically significant peak from Al is present;

- c) the Mn peak, if present, is small.

NOTE Depending on the composition of any adjacent or attached particles, other peaks can also be visible.

## 10 Test report

The test report shall contain at least the following information:

- a) reference to this part of ISO 22262 (ISO 22262-1:2012);
- b) the identification of the sample, including the location (if known by the analyst);
- c) the date of the analysis;
- d) the identity of the analyst;
- e) all applicable specimen preparation details;
- f) any procedure used not specified in this part of ISO 22262 or regarded as an optional procedure;
- g) the variety or varieties of asbestos detected;
- h) the analytical method used to identify the asbestos.

Items i) to k) shall be recorded in the laboratory data, but the extent to which they are included as part of the test report is optional:

- i) the observations made to confirm the identification of the asbestos varieties reported, including any optional procedures;
- j) the estimated mass fraction(s) of the asbestos varieties detected in ranges as follows:
  - 1) none detected,
  - 2) detected,
  - 3) 0,1 % to 5 %,
  - 4) 5 % to 50 %,
  - 5) 50 % to 100 %;

NOTE 1 These categories for reporting asbestos mass fractions are estimates only; they are intended to provide guidance in the interpretation of results. If it is necessary to make critical decisions on the basis of results in the range from "non-detected" to 5 %, sample analysis by a quantitative method is appropriate (e.g. using ISO 22262-2).

NOTE 2 The reporting category "detected" provides the analyst with a means of reporting the result when only one or two fibres are detected in the analysis, the observation of which may be a consequence of unintended contamination of the sample.

- k) the variety or varieties of any non-asbestos fibres detected, and the observations made which allowed these fibres to be discriminated from asbestos fibres.

An example of a suitable format for the test report is shown in Annex H.

## Annex A (normative)

### Types of commercial asbestos-containing material

The properties of asbestos such as non-flammability, chemical stability, and high strength have led worldwide to a broad use of this mineral in the building and industrial sectors. Asbestos–cement products, asbestos-containing lightweight panels and fire-prevention panels, asbestos packings and asbestos cloths, asbestos boards, asbestos foams, asbestos-containing fireproofing and acoustic and decorative plasters (sprayed asbestos), and asbestos-containing compositions for trowel application and putties are the most important uses. In addition, there is also a variety of products to which asbestos fibres were frequently added at smaller mass fractions, e.g. paints for protective coatings, adhesives, plastic sheets, and tiles.

Table A.1 gives the most important asbestos-containing materials with examples of their applications and the typical asbestos mass fractions. In exceptional cases, asbestos mass fractions deviating from those quoted may have been used.

**Table A.1 — Asbestos-containing materials; examples of use and typical asbestos content**

Product	Examples of application	Typical asbestos type and mass fraction
Asbestos–cement flat boards	Roof claddings Sidings Banister elements Windowsills Staircases Partition walls Support for cable runs In small sizes as slates and shingles in the roofing and siding sectors	Chrysotile 10 %–12 %, Sometimes also <5 % crocidolite or amosite in addition to chrysotile
Asbestos–cement corrugated sheets	Roof claddings Perimeter insulation Sidings in the industrial sector	Chrysotile 10 %–12 %, sometimes also, with some manufacturers, <5 % crocidolite in addition to chrysotile
Asbestos–cement pipes or ducts	Drinking water and wastewater pipes Service pipes Inlet air and exhaust air ducts Cable shafts	Chrysotile 10 %–15 %. Drinking water pipe also <5 % crocidolite or amosite in addition to chrysotile
Asbestos–cement mouldings	Standard ashtrays Flower boxes Garden articles Sculptures	Chrysotile 10 %–12 %

Table A.1 (continued)

Product	Examples of application	Typical asbestos type and mass fraction
Asbestos-containing lightweight building boards or fire-resistant panels	Sealing of openings in walls required to be fire resistant Fire-protection encasement of ventilation ducts, cable ducts and cable shafts Fire closures in walls required to be fire resistant (fire shutters, fire barriers) Fire-protection encasements Smoke-removal ducts Insert in fire-resistant doors and gates Substructure of luminaries (lighting fixtures)	Chrysotile ~15 % and amosite ~15 %
Asbestos-containing lightweight building boards or fire-resistant panels	Lining fire-hazard rooms Partition walls, partition surfaces, doors Sanitary modules Support and beam encasements Smoke aprons Fire locks	Chrysotile <50 %, sometimes amosite <35 %
Asbestos-containing pipe and boiler insulations	Corrugated paper pipe insulation 85 % magnesia block and pipe insulation Calcium silicate block and pipe insulation	Chrysotile 30 %–100 % Total of 15 % asbestos, can be chrysotile, amosite or crocidolite, or any mixture of two or more.
Asbestos packing, asbestos cloth	Seals or sealing strips on lightweight walls required to be fire resistant (at ceiling, floor, joints between elements, wall terminations) Seals on pipe and duct feed-throughs in walls and ceilings Seals between flanges of ventilation ducts Seals on fire-resistant glazing, shelter doors, chimney soot doors Seals and insulation on heat-generation systems, hot pipes and hot valves Fire blankets Heat-resistant clothing, heat-resistant gloves Lining of pipe clips for hot water, steam and sprinkler pipes Lamp wicks Mantles for gas lamps	Predominantly chrysotile (80 %–100 %); crocidolite for acid-resistant applications
Asbestos millboards	Sealing strips on lightweight walls required to be fire resistant (at ceiling, floor, joints between elements, wall terminations) Substructure of luminaries (lighting fixtures) Bottom coating of wooden windowsills over radiators	Chrysotile 80 %–100 %
Asbestos foams	Infilling (sealing) of movement joints Seals at fire shutters and fire barriers	Chrysotile ~50 %

**Table A.1** (continued)

Product	Examples of application	Typical asbestos type and mass fraction
Sprayed asbestos	<p>Contour-following fire-resistant coating of steel structures</p> <p>Coating of ceilings and walls in music auditoria, theatres, churches, garages, industrial rooms (for noise protection)</p> <p>Sealing off openings for cable, pipe and duct feed-throughs through walls required to be fire resistant</p> <p>Encasing of ventilation ducts</p>	<p>Chrysotile, crocidolite or amosite 40 %–70 %, also mixtures of mineral wool with either 20 % amosite or up to 30 % chrysotile. Other mixtures include 15 % chrysotile with either perlite or vermiculite, and gypsum.</p> <p>Sprayed vermiculite coatings (with or without chrysotile) can contain up to 2 % tremolite, some of which can be asbestiform.</p> <p>Several per cent of tremolite asbestos (Japan)</p>
Sprayed decorative coatings (texture coats)	Coating of ceilings and walls to provide a textured surface which masks irregularities	Chrysotile <5 %. Some constituents can also contain tremolite. Some of the tremolite can be asbestiform.
Gypsum wallboard joint compounds	Provides smooth joint between adjacent panels	Chrysotile <5 %. Some constituents can also contain low mass fractions of tremolite.
Asbestos-containing troweled-on compositions and putty	<p>Grouting of prefabricated concrete components</p> <p>Sealing of movement joints</p> <p>Pipe feeds through walls and ceilings</p> <p>Door casings of fire-resistant doors</p> <p>Anti-drumming coatings (car preservation)</p> <p>Coating of underwater structures</p> <p>Baseboard coating on house walls</p>	Chrysotile <20 %
Asbestos-containing floorings	<p>Reinforcement in flexible sheets</p> <p>Rot-resistant support layer as underlay of cushion PVC flooring materials</p>	<p>Chrysotile 10 %–20 %</p> <p>Chrysotile 80 %–100 %</p>
Asphalt or PVC asbestos floor tiles	Reinforcement	Asphalt tiles containing chrysotile <35 %, PVC tiles containing chrysotile <20 %
Rubberized asbestos seals	Gaskets for pipe flanges	Chrysotile 50 %–90 %
Asbestos-containing friction products	<p>Brake linings</p> <p>Brake bands</p> <p>Clutch linings</p>	Chrysotile 10 %–70 %
Acid-resistant containers	<p>Lead-acid battery boxes</p> <p>Drums for acid</p>	Crocidolite 10 %–50 %
Filter media	<p>Air filters</p> <p>Liquid filters</p> <p>Sterile and aseptic filters</p> <p>Clarifying sheets</p> <p>Diaphragms for chloralkali electrolysis processes</p> <p>Filtration media for Gooch crucibles</p>	<p>Chrysotile, rarely amosite 95 %</p> <p>For Gooch crucibles, 100 % tremolite or anthophyllite</p>



Table A.1 (continued)

Product	Examples of application	Typical asbestos type and mass fraction
Talc (asbestos content dependent on deposit)	Release agents for electric cables, rubber products Release agents in the confectionery industry Tailor's chalk Paper manufacture Medicine, cosmetics	Chrysotile and/or actinolite/tremolite. Some of the actinolite/tremolite can be asbestiform
Vermiculite (exfoliated)	Attic and wall cavity insulation Fireproofing Horticultural products	Depends on the source of the vermiculite. Vermiculite from Montana, USA, can contain up to 6 % of a mixture of amphibole types, some of which can be asbestiform
Industrial minerals: wollastonite, sepiolite, attapulgite	Ceramics manufacture Plastics fillers Surfacing materials and joint compounds Ceiling tiles Drilling muds (attapulgite)	Depends on the source of the mineral. Can contain several per cent of tremolite or actinolite, some of which can be asbestiform.
Industrial minerals: calcite, dolomite and gypsum	Manufacture of building materials Industrial uses	Depends on the source of the mineral. Carbonate minerals can contain several per cent of tremolite or actinolite, some of which can be asbestiform
Industrial minerals: mica	Ceramics manufacture Manufacture of building materials	Depends on the source of the mineral. Can contain tremolite or actinolite, some of which can be asbestiform
Asphalt surfacings	Road construction	Chrysotile, generally $\leq 1$ %
Wall and ceiling plasters	Interior wall and ceiling coatings, with or without aggregate and fibres such as animal hair or jute	Chrysotile. Generally locally mixed and inhomogeneous. Can be any mass fraction up to approximately 3 %
Drilling muds	Oil exploration, rock drilling	Chrysotile. Often the chrysotile is very fine and short, sometimes originating from Coalinga, California. Can contain <100 % chrysotile
Chemical products for construction, and other products	Bitumen, roofing and sealing sheets Sealing putties Glazing putties Bituminous coatings Fillers and sealers Jointing compounds Paints Glues Fire retardants Sub-floor protection	Chrysotile <30 % Chrysotile <2 % Chrysotile <4 % Chrysotile <30 % Chrysotile <25 % Chrysotile <5 % Chrysotile <9 % Chrysotile <4 % Chrysotile <10 % Chrysotile <4 %

## Annex B (normative)

### Interference colour chart

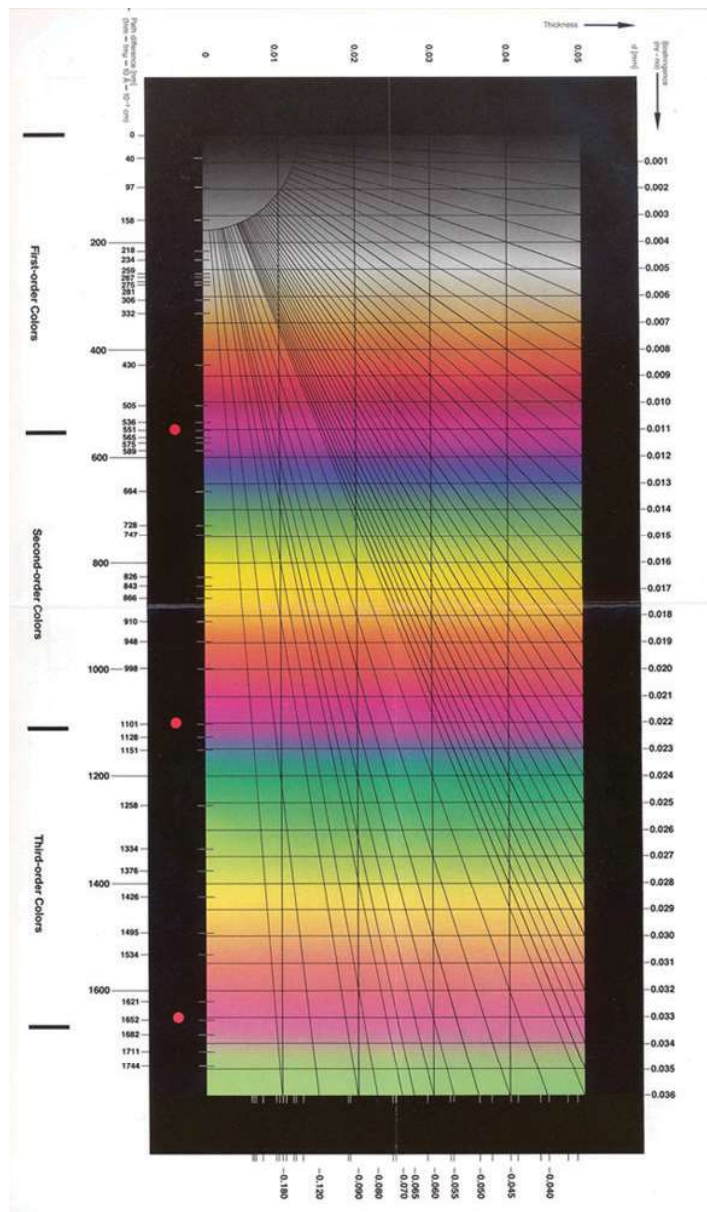


Figure B.1 — Interference colour chart

## Annex C (normative)

### Dispersion staining charts

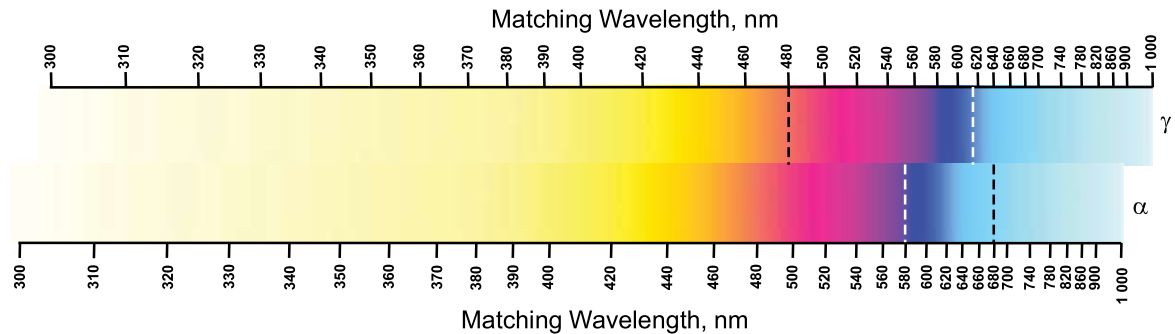


Figure C.1 — Central stop dispersion staining colours for chrysotile in 1,550 RI liquid

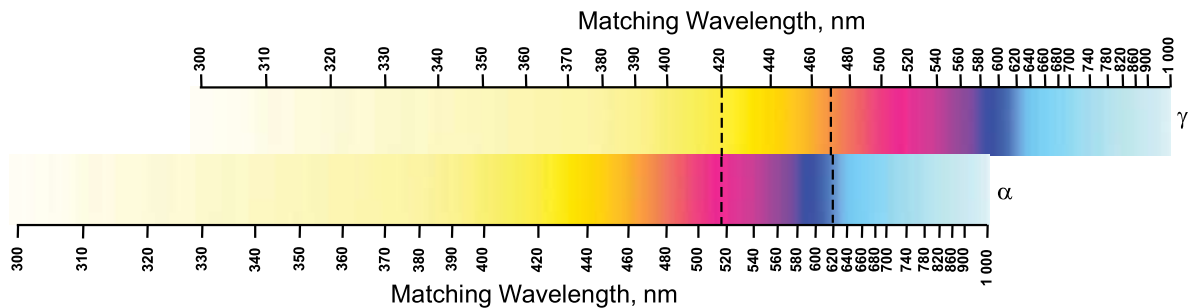


Figure C.2 — Central stop dispersion staining colours for amosite in 1,680 RI liquid

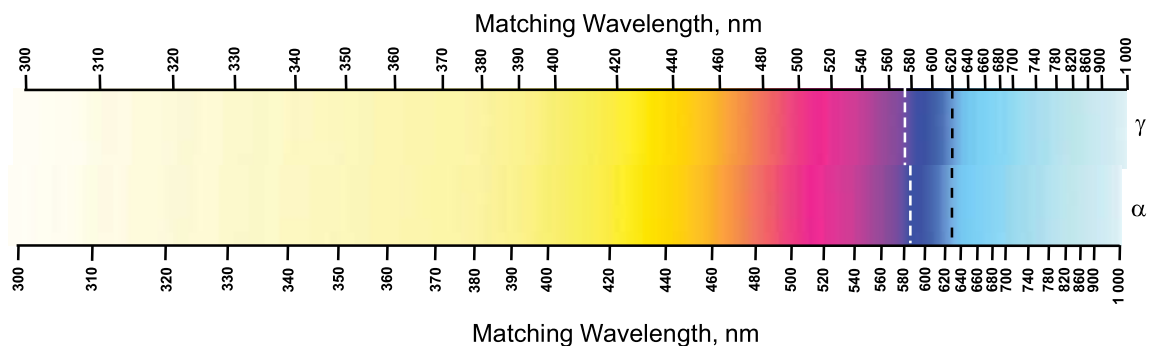


Figure C.3 — Central stop dispersion staining colours for crocidolite in 1,700 RI liquid

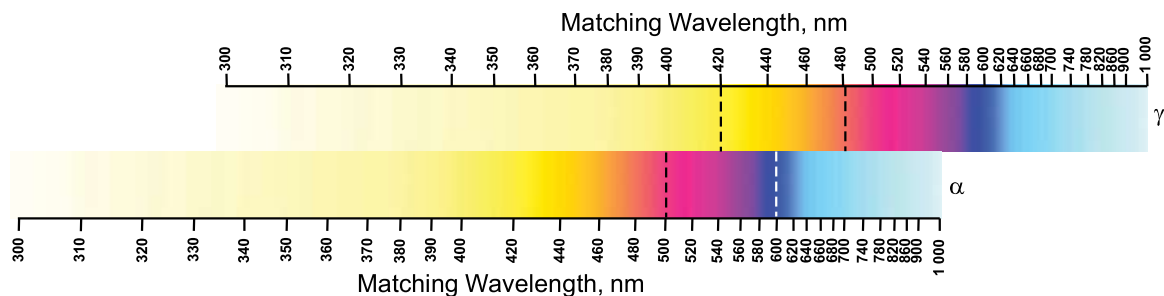


Figure C.4 — Central stop dispersion staining colours for tremolite in 1,605 RI liquid

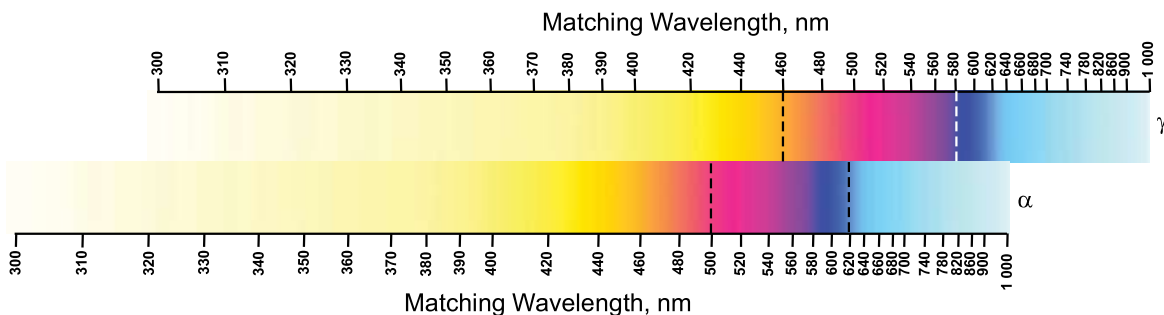


Figure C.5 — Central stop dispersion staining colours for actinolite in 1,630 RI liquid

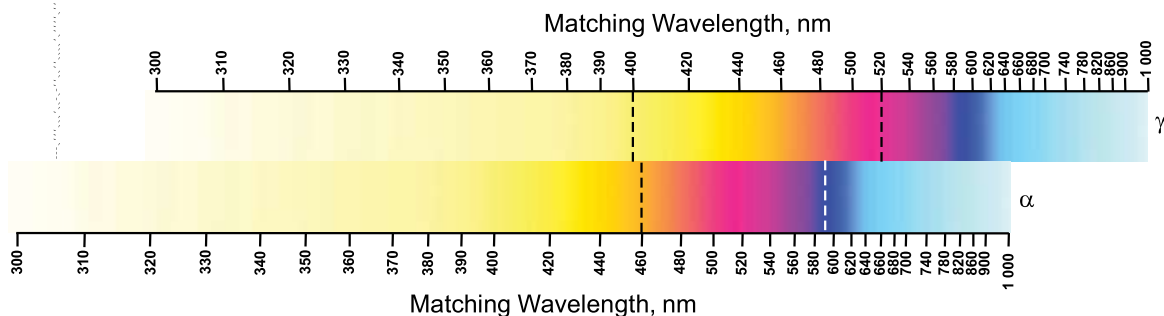


Figure C.6 — Central stop dispersion staining colours for anthophyllite in 1,605 RI liquid

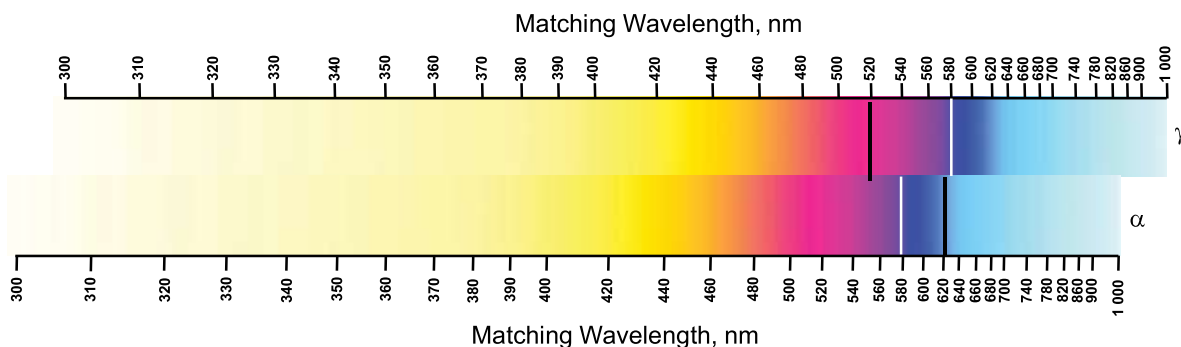


Figure C.7 — Central stop dispersion staining colours for richterite/winchite asbestos in 1,630 RI liquid

## Annex D

(normative)

### Asbestos identification by PLM and dispersion staining in commercial materials

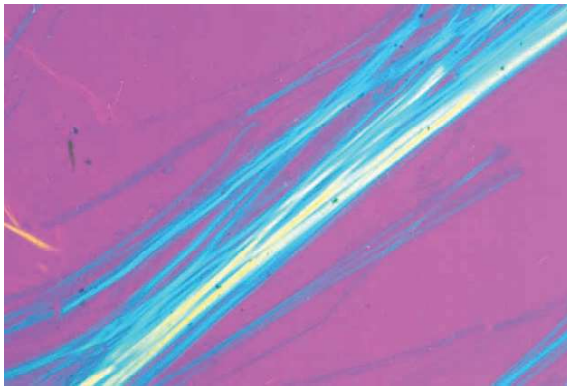


Figure D.1 — PLM micrograph of SRM 1866 chrysotile in 1,550 RI liquid — Crossed polars with 530 nm retardation plate

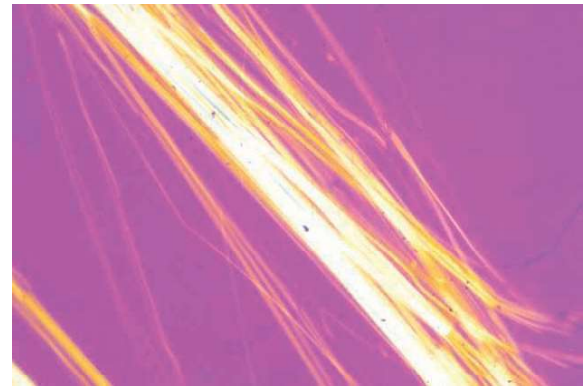


Figure D.2 — PLM micrograph of SRM 1866 chrysotile in 1,550 RI liquid — Crossed polars with 530 nm retardation plate

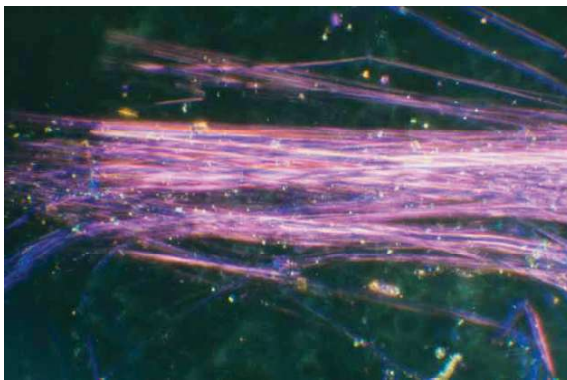


Figure D.3 — SRM 1866 chrysotile in 1,550 RI liquid viewed in dispersion staining — Fibre length parallel to polarizer vibration direction

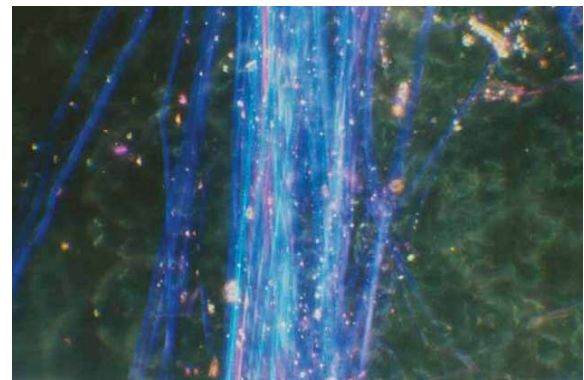
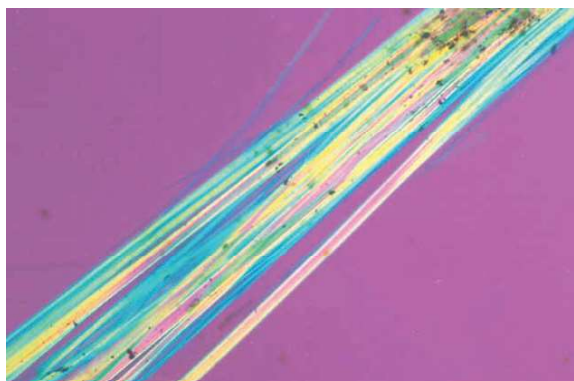
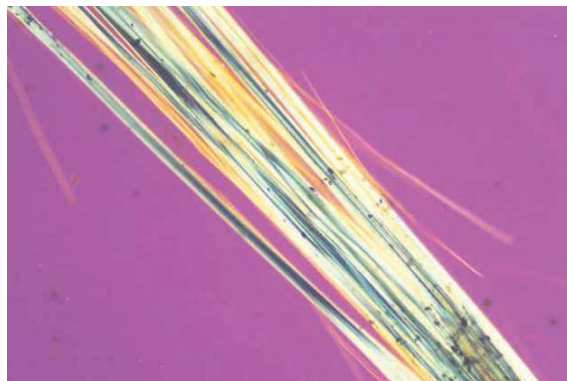


Figure D.4 — SRM 1866 chrysotile in 1,550 RI liquid viewed in dispersion staining — Fibre length normal to polarizer vibration direction

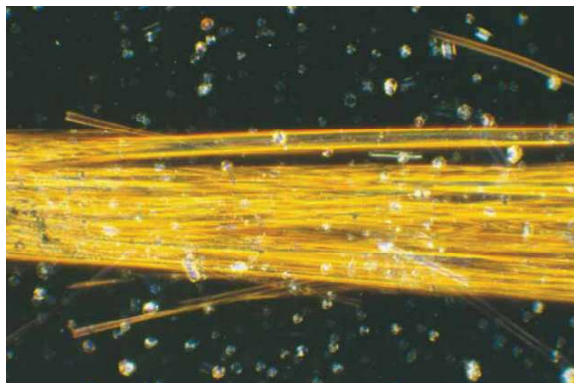




**Figure D.5 — PLM micrograph of SRM 1866 amosite in 1,680 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.6 — PLM micrograph of SRM 1866 amosite in 1,680 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.7 — SRM 1866 amosite in 1,680 RI liquid viewed in dispersion staining — Fibre length parallel to polarizer vibration direction**



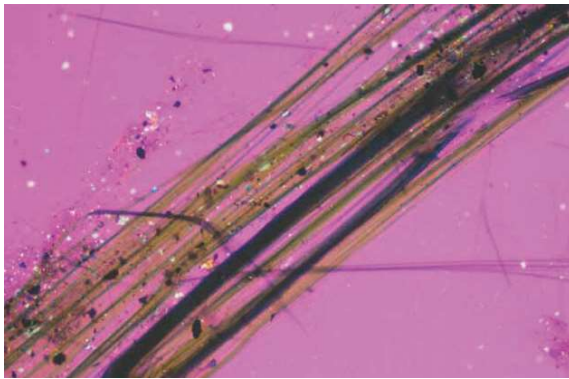
**Figure D.8 — SRM 1866 amosite in 1,680 RI liquid viewed in dispersion staining — Fibre length normal to polarizer vibration direction**



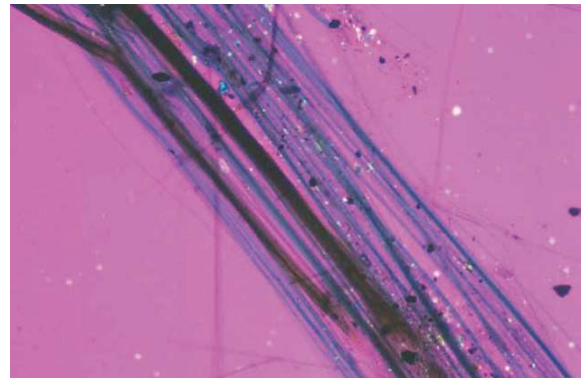
**Figure D.9 — Heated amosite in 1,680 RI liquid viewed in plane polarized light — Fibre length parallel to polarizer vibration direction**



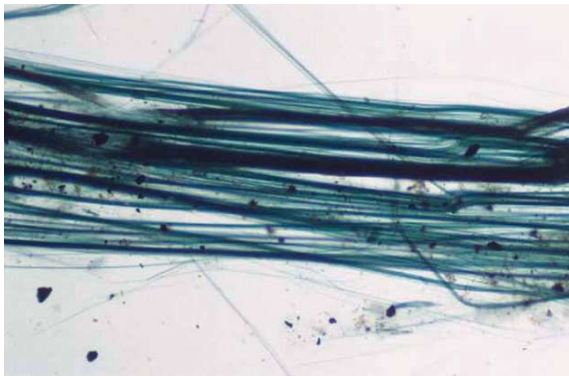
**Figure D.10 — Heated amosite in 1,680 RI liquid viewed in plane polarized light — Fibre length parallel to polarizer vibration direction**



**Figure D.11 — PLM micrograph of SRM 1866 crocidolite in 1,700 RI liquid — Crossed polars with 530 nm retardation plate**



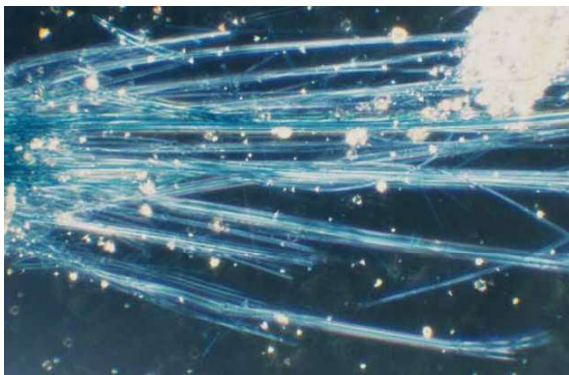
**Figure D.12 — PLM micrograph of SRM 1866 crocidolite in 1,700 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.13 — SRM 1866 crocidolite in 1,700 RI liquid in plane polarized light — Fibres parallel to polarizer vibration direction**



**Figure D.14 — SRM 1866 crocidolite in 1,700 RI liquid in plane polarized light — Fibres normal to polarizer vibration direction**

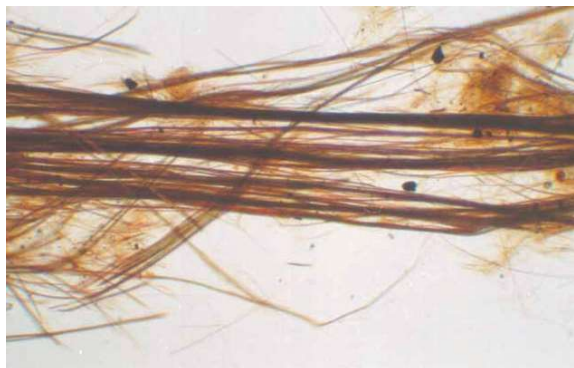


**Figure D.15 — SRM 1866 crocidolite in 1,700 RI liquid — Dispersion staining — Fibre lengths parallel to polarizer vibration direction**



**Figure D.16 — SRM 1866 crocidolite in 1,700 RI liquid — Dispersion staining — Fibre lengths normal to polarizer vibration direction**





**Figure D.17 — Heated crocidolite viewed in plane polarized light — Fibre length parallel to polarizer vibration direction**



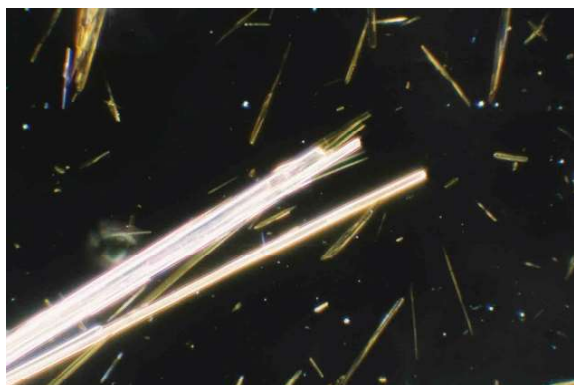
**Figure D.18 — Heated crocidolite viewed in plane polarized light — Fibre length normal to polarizer vibration direction**



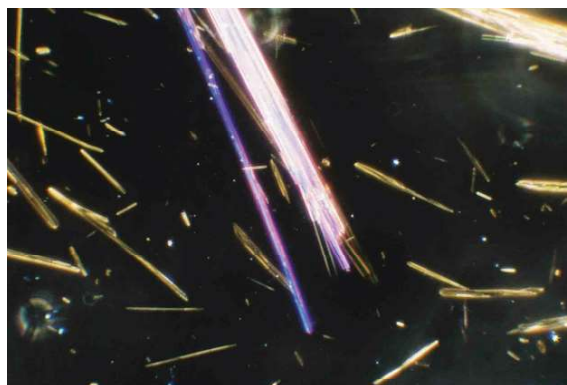
**Figure D.19 — PLM micrograph of SRM 1867 tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.20 — PLM micrograph of SRM 1867 tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**

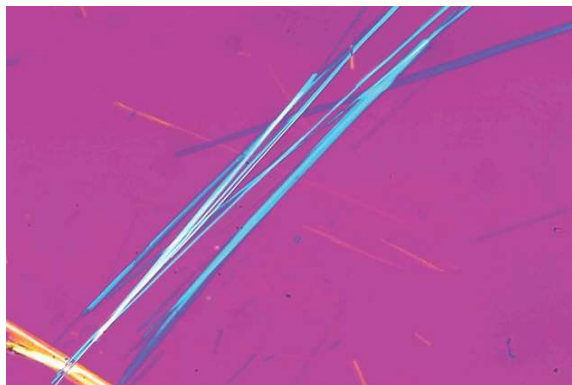


**Figure D.21 — SRM 1867 tremolite in 1,605 RI liquid viewed in dispersion staining — Fibres at extinction position**

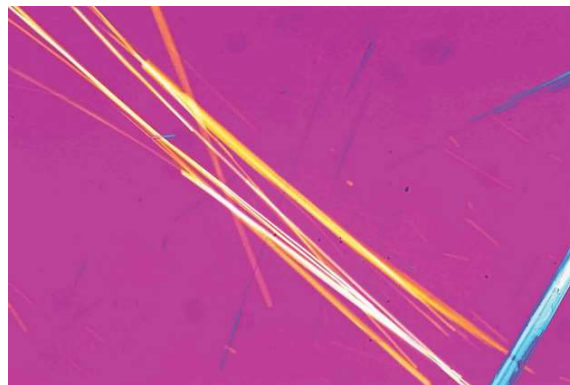


**Figure D.22 — SRM 1867 tremolite in 1,605 RI liquid viewed in dispersion staining — Fibres at extinction position**

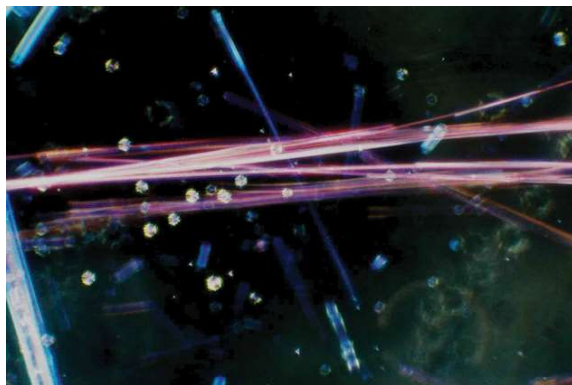




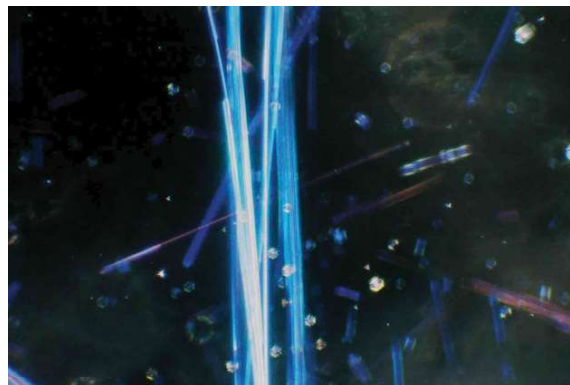
**Figure D.23 — PLM micrograph of SRM 1867 tremolite in 1,625 RI liquid — Crossed polars with 530 nm retardation plate**



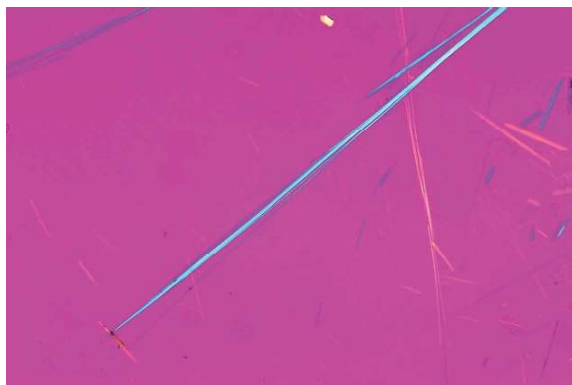
**Figure D.24 — PLM micrograph of SRM 1867 tremolite in 1,625 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.25 — SRM 1867 tremolite in 1,625 RI liquid viewed in dispersion staining — Fibres at extinction position**



**Figure D.26 — SRM 1867 tremolite in 1,625 RI liquid viewed in dispersion staining — Fibres at extinction position**



**Figure D.27 — PLM micrograph of SRM 1867 actinolite in 1,630 RI liquid — Crossed polars with 530 nm retardation plate**



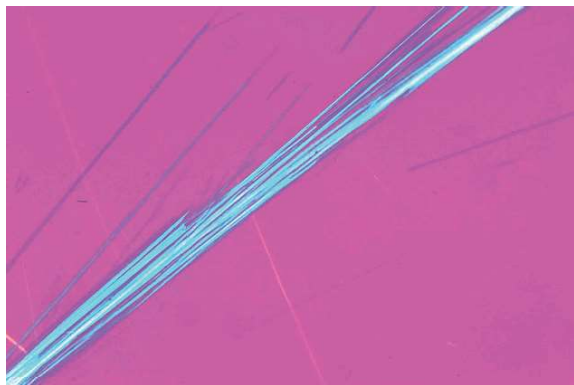
**Figure D.28 — PLM micrograph of SRM 1867 actinolite in 1,630 RI liquid — Crossed polars with 530 nm retardation plate**



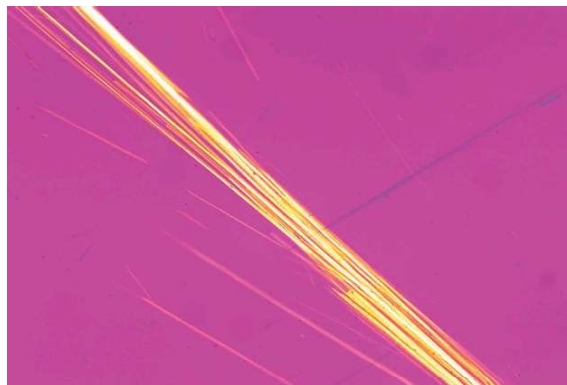
**Figure D.29 — SRM 1867 actinolite in 1,630 RI liquid viewed in dispersion staining — Purple fibre at extinction position**



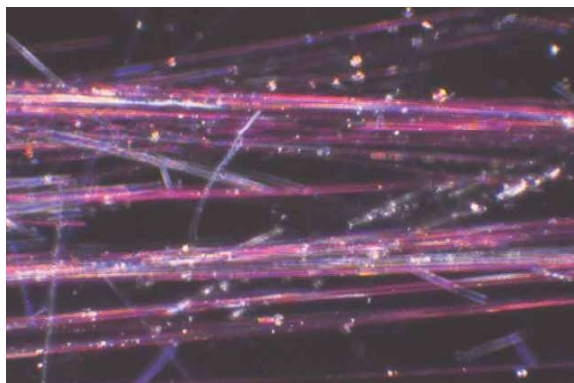
**Figure D.30 — SRM 1867 actinolite in 1,630 RI liquid viewed in dispersion staining — Light blue fibre at extinction position**



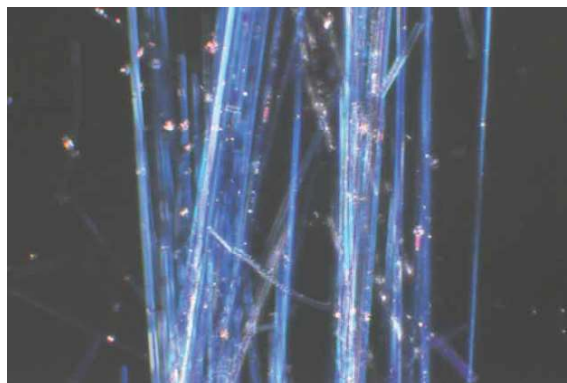
**Figure D.31 — PLM micrograph of SRM 1867 anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



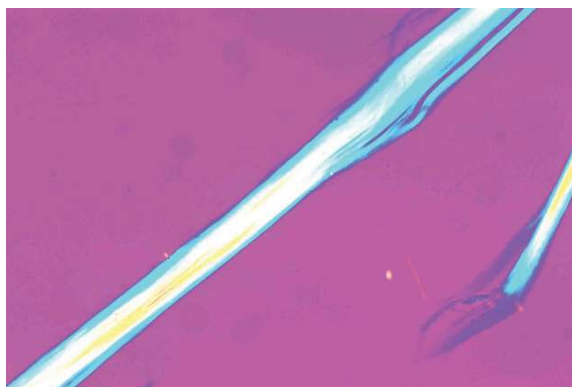
**Figure D.32 — PLM micrograph of SRM 1867 anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



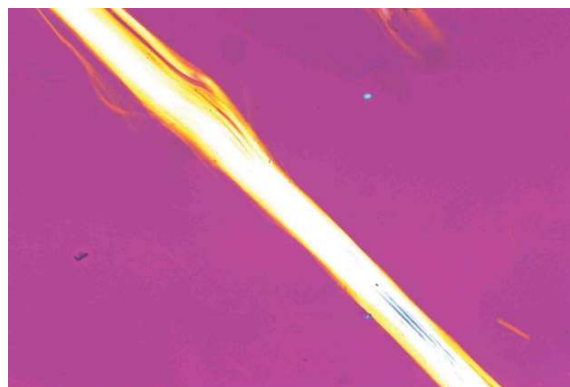
**Figure D.33 — SRM 1867 anthophyllite in 1,630 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction**



**Figure D.34 — SRM 1867 anthophyllite in 1,630 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction**



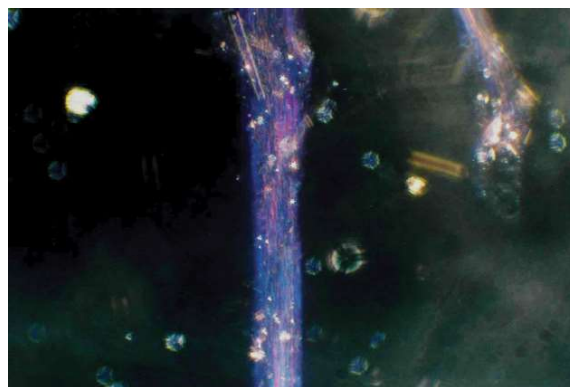
**Figure D.35 — PLM micrograph of HSE tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



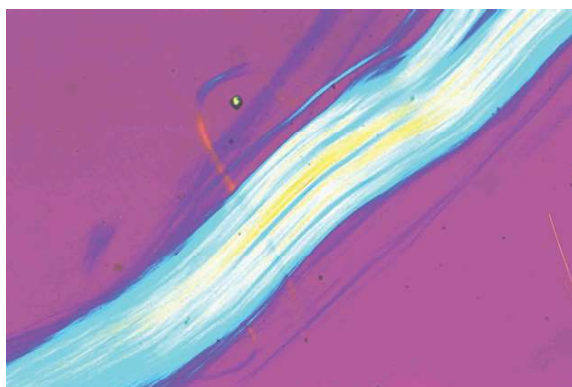
**Figure D.36 — PLM micrograph of HSE tremolite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



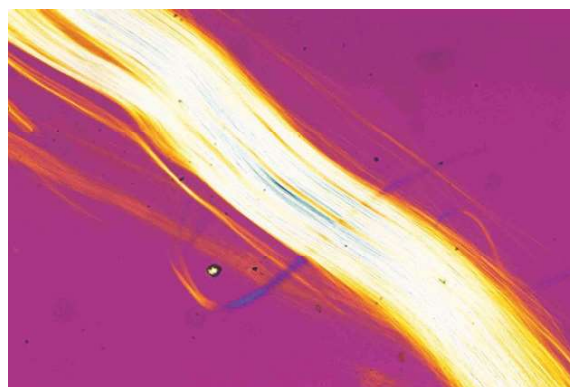
**Figure D.37 — HSE tremolite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction**



**Figure D.38 — HSE tremolite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction**

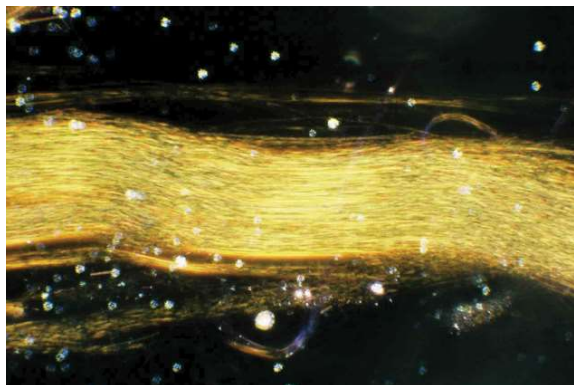


**Figure D.39 — PLM micrograph of HSE actinolite in 1,640 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.40 — PLM micrograph of HSE actinolite in 1,640 RI liquid — Crossed polars with 530 nm retardation plate**

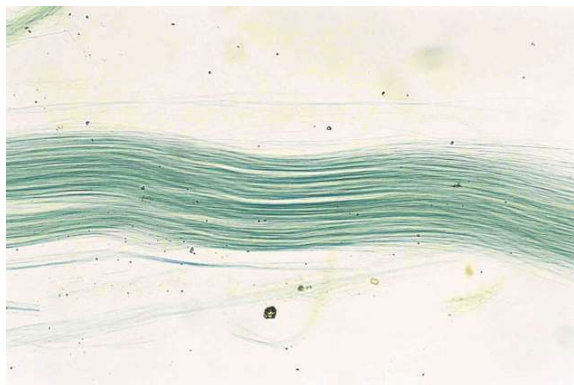




**Figure D.41 — HSE actinolite in 1,640 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction**



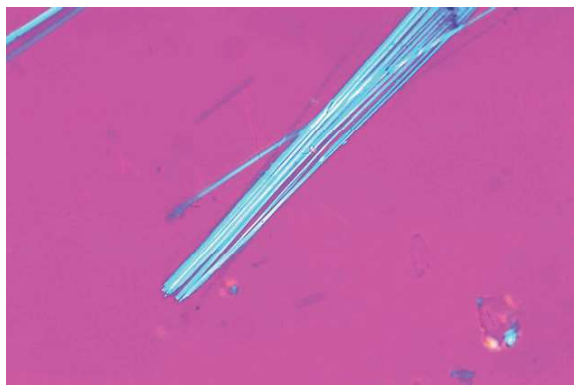
**Figure D.42 — HSE actinolite in 1,640 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction**



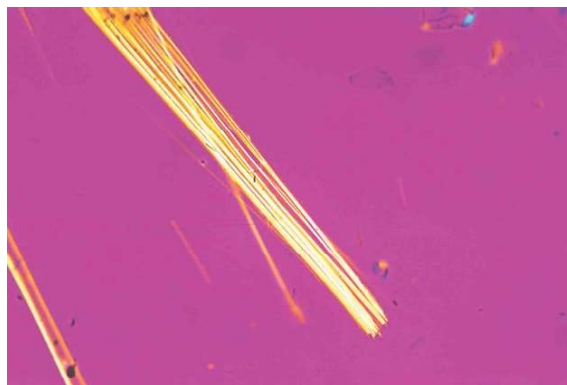
**Figure D.43 — HSE actinolite in 1,640 RI liquid in plane polarized light — Fibres parallel to polarizer vibration direction**



**Figure D.44 — HSE actinolite in 1,640 RI liquid in plane polarized light — Fibres normal to polarizer vibration direction**



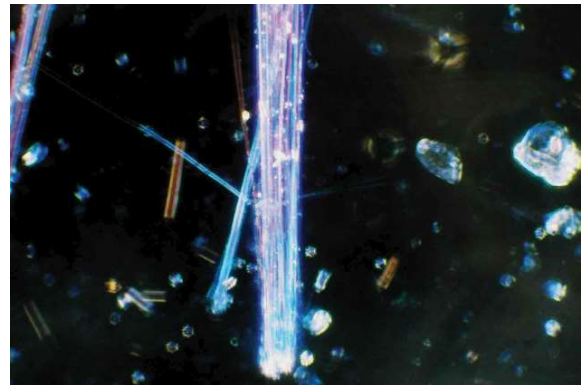
**Figure D.45 — PLM micrograph of HSE anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



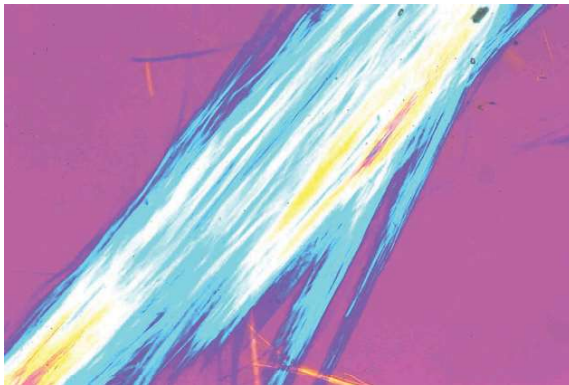
**Figure D.46 — PLM micrograph of HSE anthophyllite in 1,605 RI liquid — Crossed polars with 530 nm retardation plate**



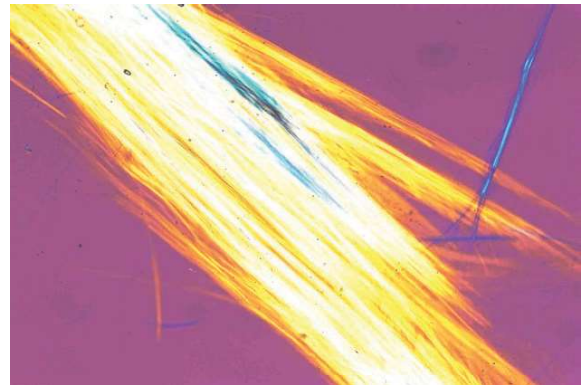
**Figure D.47 — HSE anthophyllite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths parallel to polarizer vibration direction**



**Figure D.48 — HSE anthophyllite in 1,605 RI liquid viewed in dispersion staining — Fibre lengths normal to polarizer vibration direction**



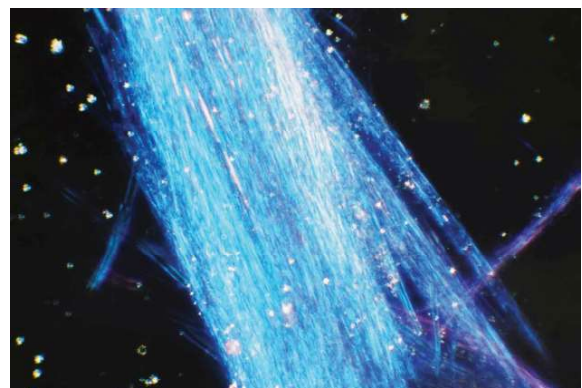
**Figure D.49 — PLM micrograph of richterite/winchite asbestos in 1,630 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.50 — PLM micrograph of richterite/winchite asbestos in 1,630 RI liquid — Crossed polars with 530 nm retardation plate**



**Figure D.51 — Richterite/winchite asbestos in 1,630 RI liquid viewed in dispersion staining — Fibres at extinction position**



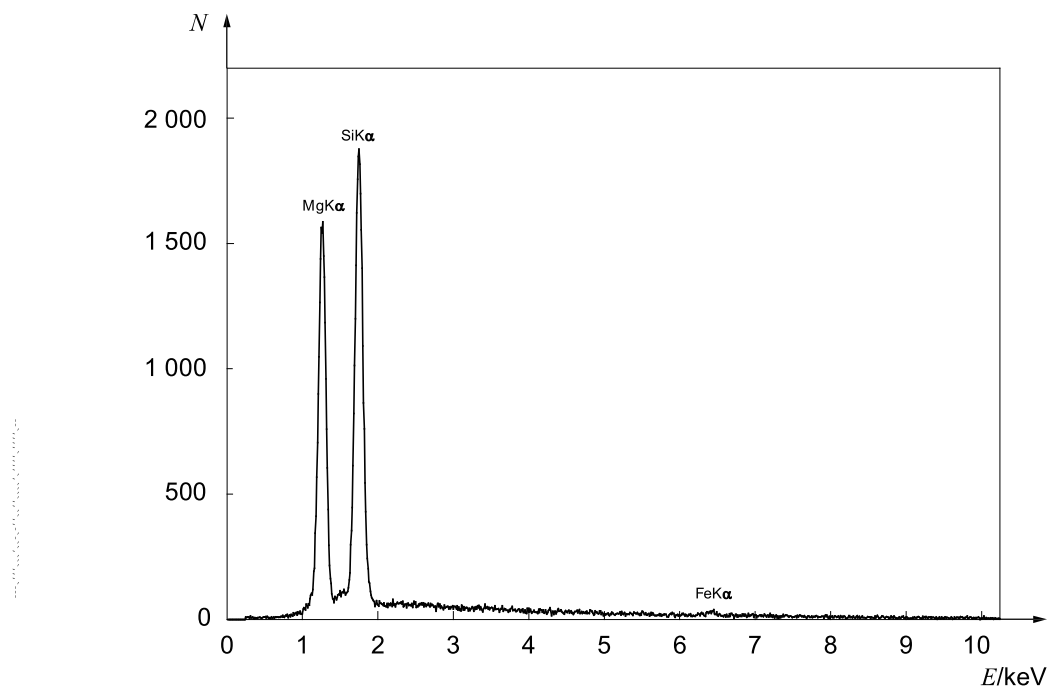
**Figure D.52 — Richterite/winchite asbestos in 1,630 RI liquid viewed in dispersion staining — Fibres at extinction position**

## Annex E (normative)

### Asbestos identification by SEM in commercial materials

Figures E.1 to E.11 are examples of EDXA spectra collected on an SEM operating at 15 kV and using a silicon solid-state detector with a beryllium window. The SEM specimens were prepared by mounting representative fibre bundles from SRM 1866, SRM 1867, and the HSE reference asbestos varieties on adhesive tape on SEM specimen stubs. All specimens were carbon coated in a vacuum evaporator.

Prior to use of this part of ISO 22262, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific X-ray detector.



#### Key

$N$  counts       $E$  X-ray energy

**Figure E.1 — Energy dispersive X-ray spectrum obtained from SRM 1866 chrysotile**

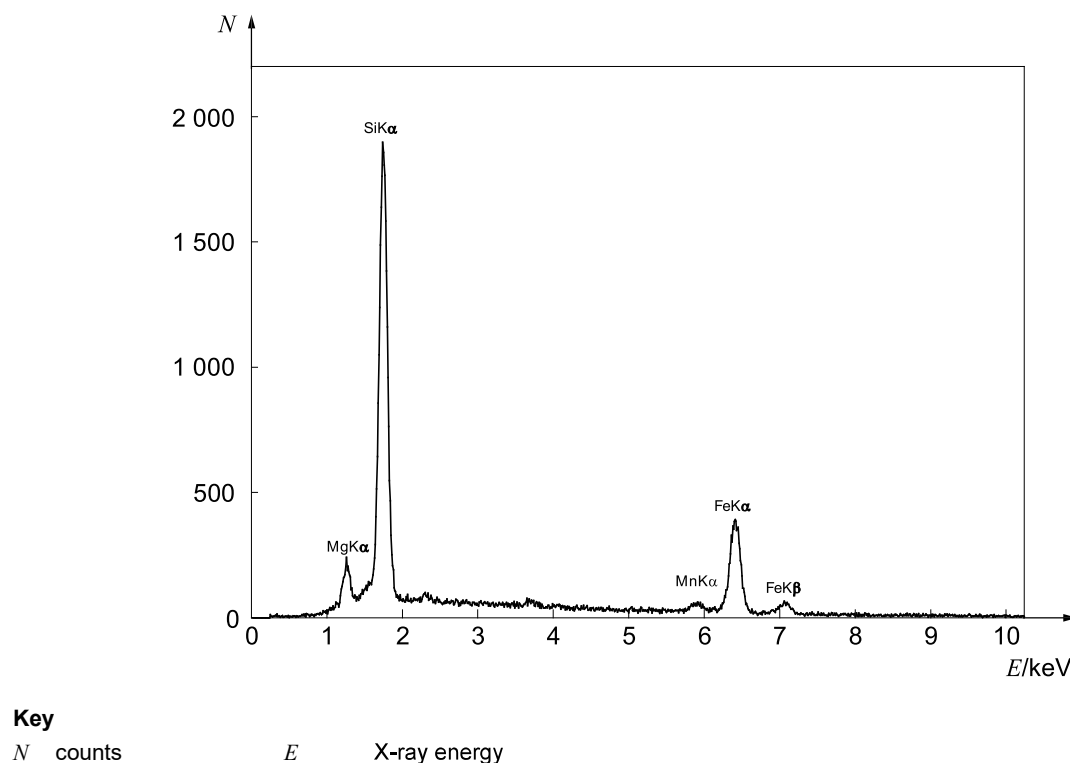


Figure E.2 — Energy dispersive X-ray spectrum obtained from SRM 1866 amosite

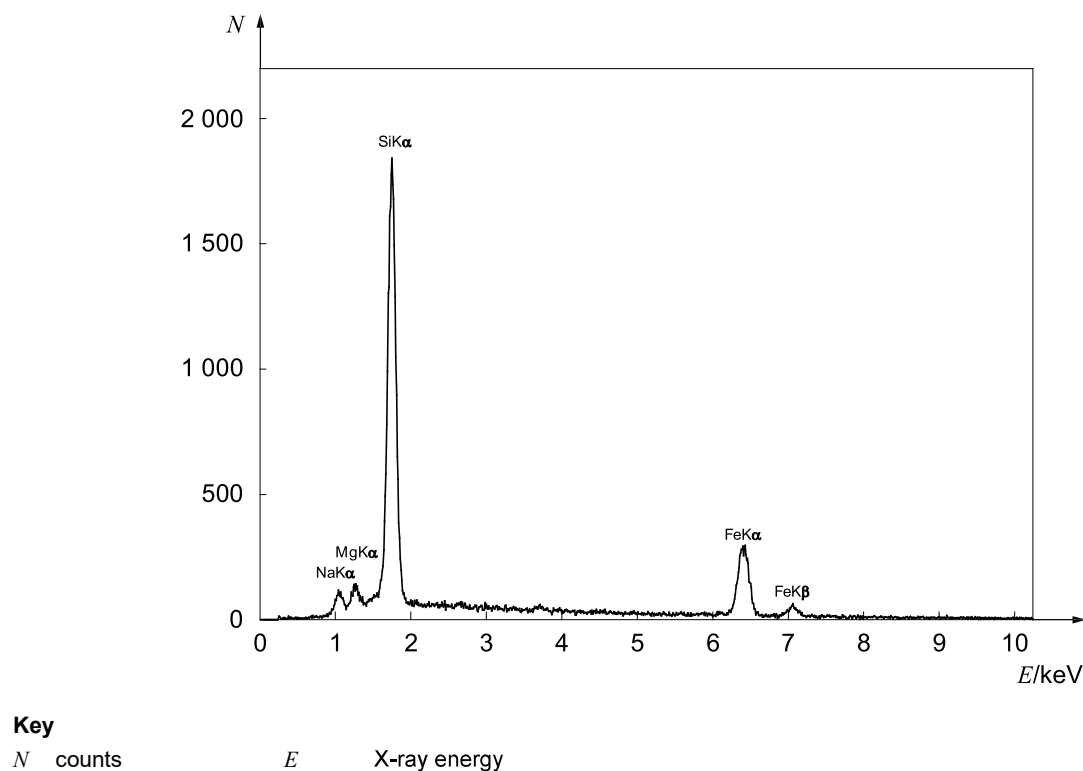
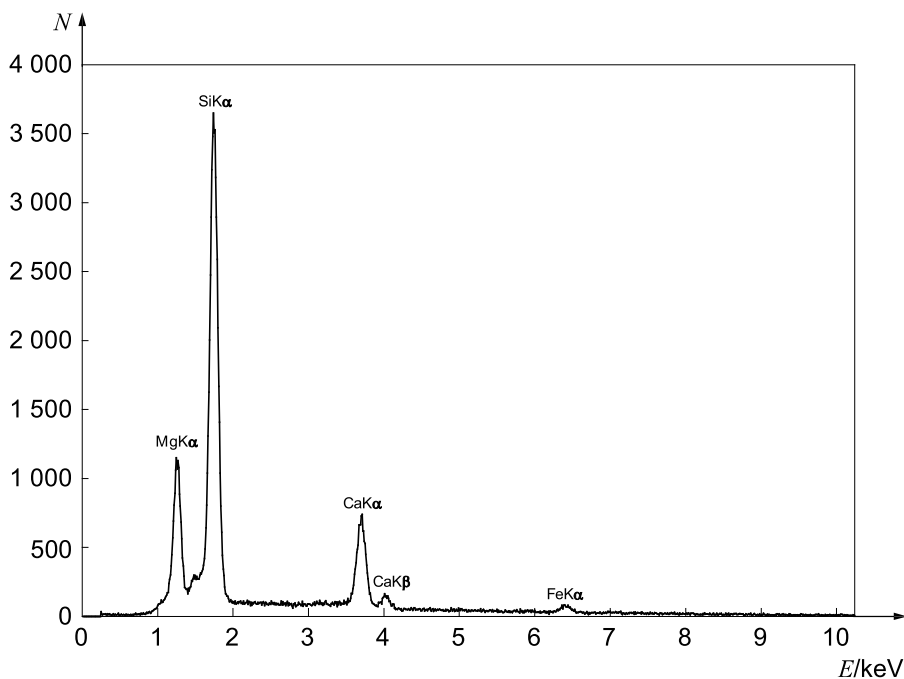


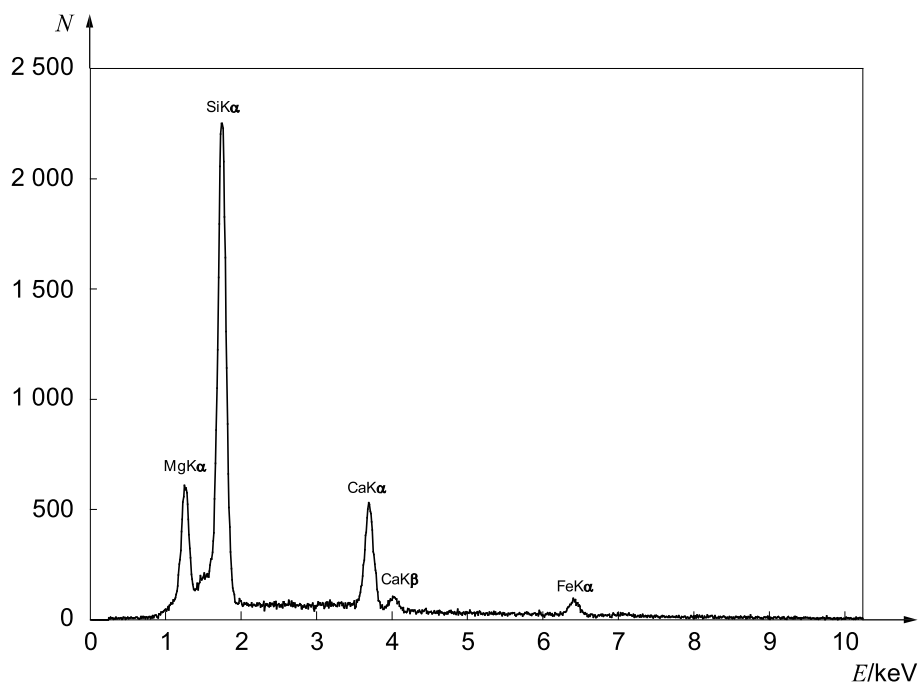
Figure E.3 — Energy dispersive X-ray spectrum obtained from SRM 1866 crocidolite



**Key**

$N$  counts       $E$  X-ray energy

**Figure E.4 — Energy dispersive X-ray spectrum obtained from SRM 1867 tremolite**



**Key**

$N$  counts       $E$  X-ray energy

**Figure E.5 — Energy dispersive X-ray spectrum obtained from SRM 1867 actinolite**



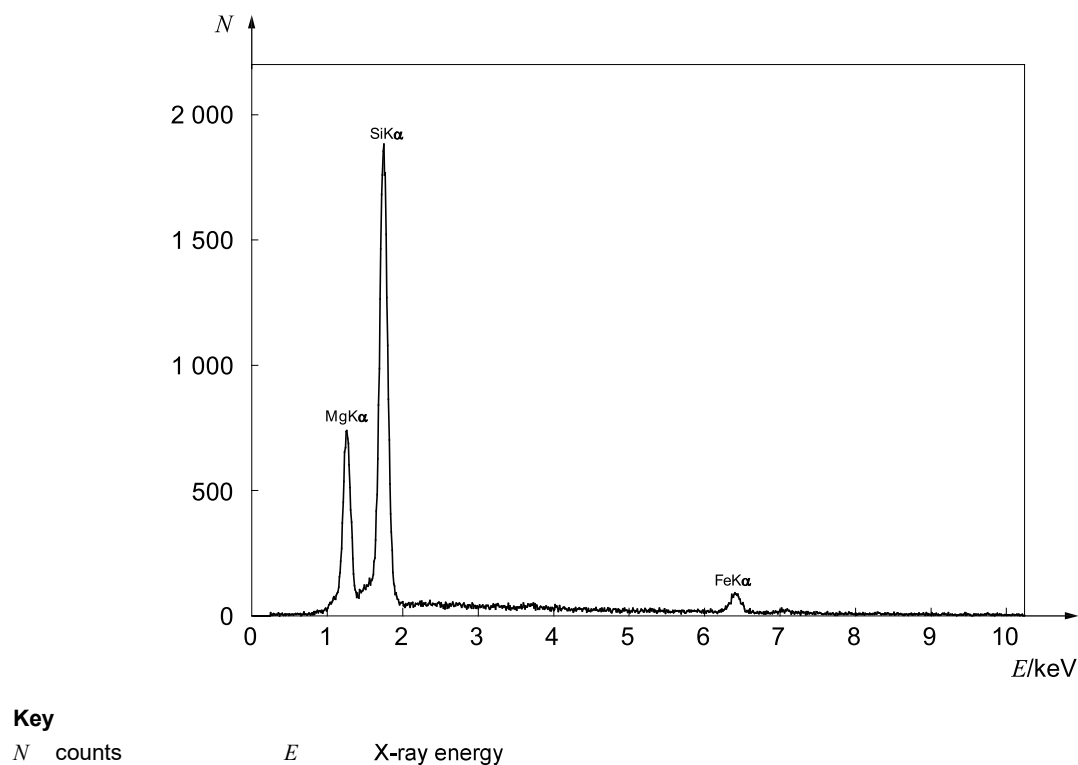


Figure E.6 — Energy dispersive X-ray spectrum obtained from SRM 1867 anthophyllite

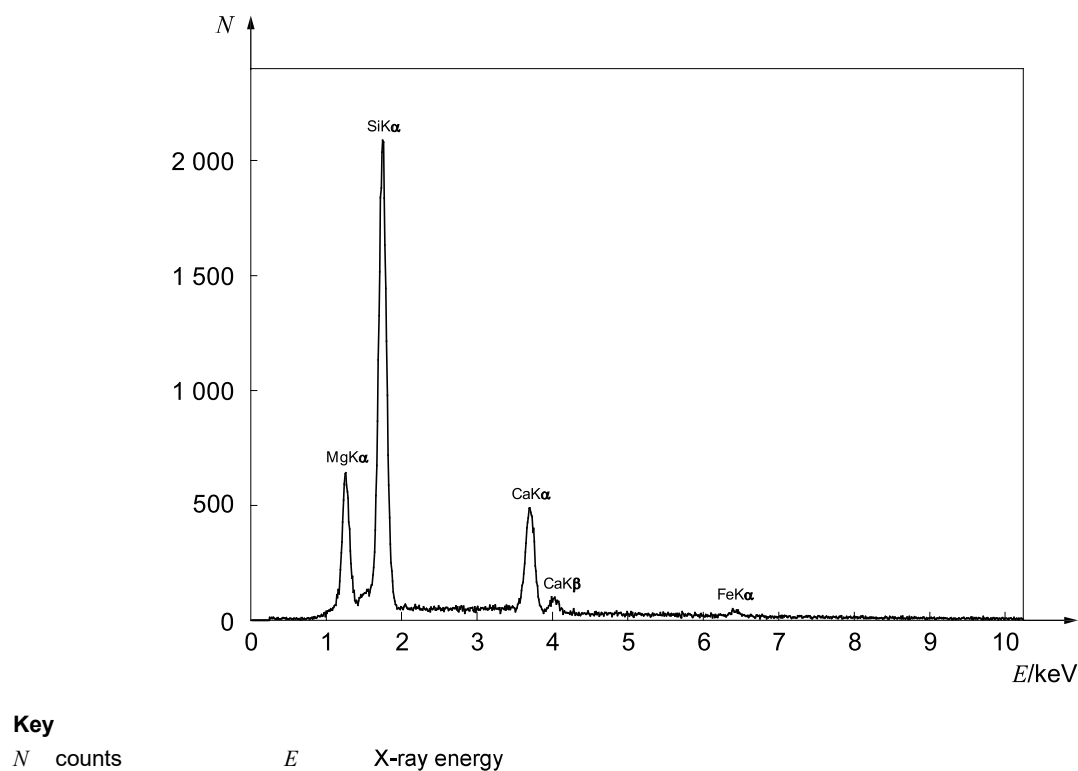
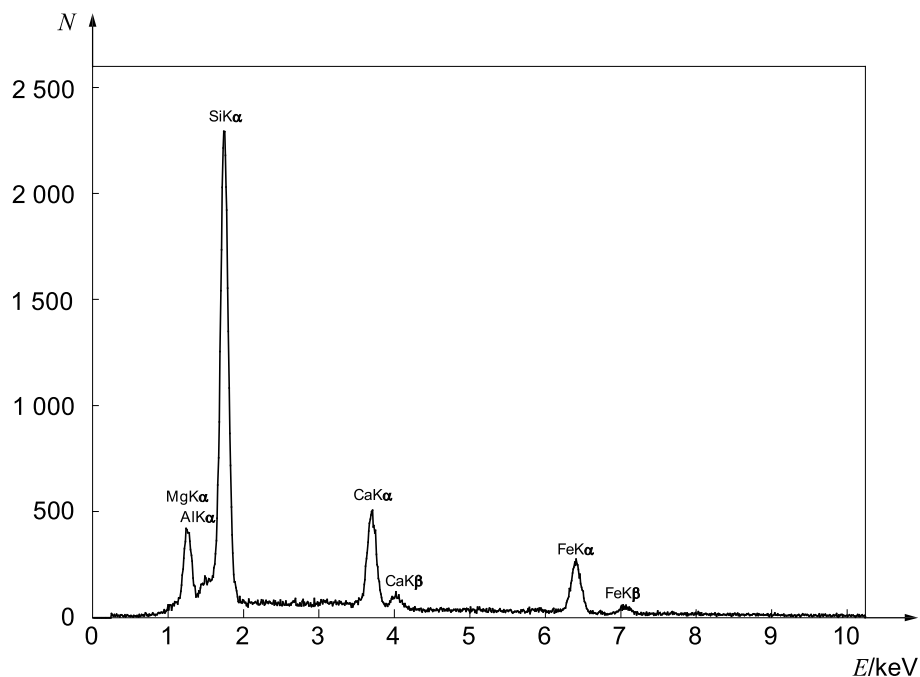


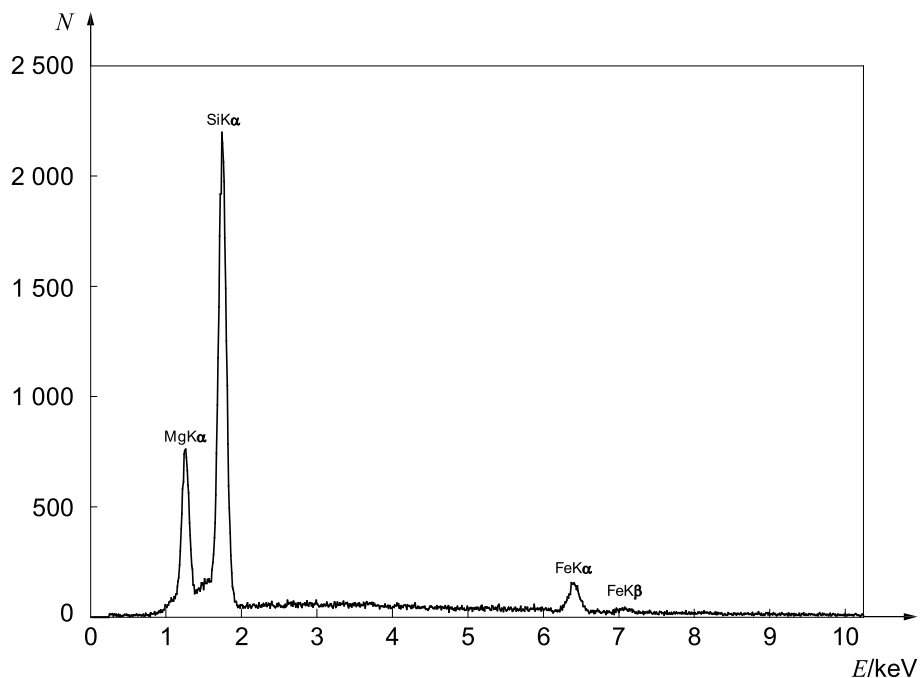
Figure E.7 — Energy dispersive X-ray spectrum obtained from HSE tremolite



**Key**

$N$  counts  $E$  X-ray energy

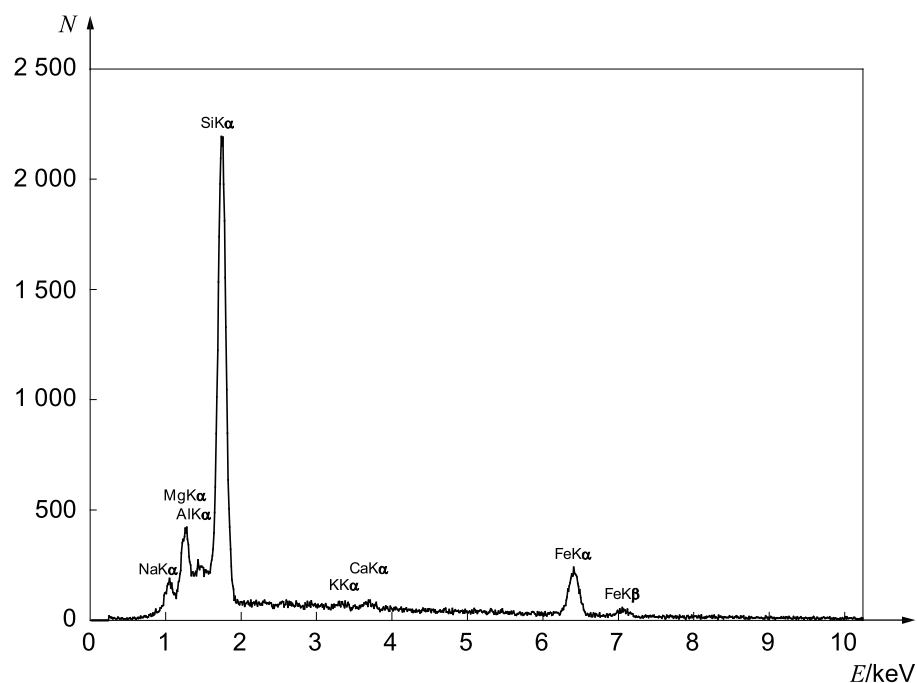
**Figure E.8 — Energy dispersive spectrum obtained from HSE actinolite**



**Key**

$N$  counts  $E$  X-ray energy

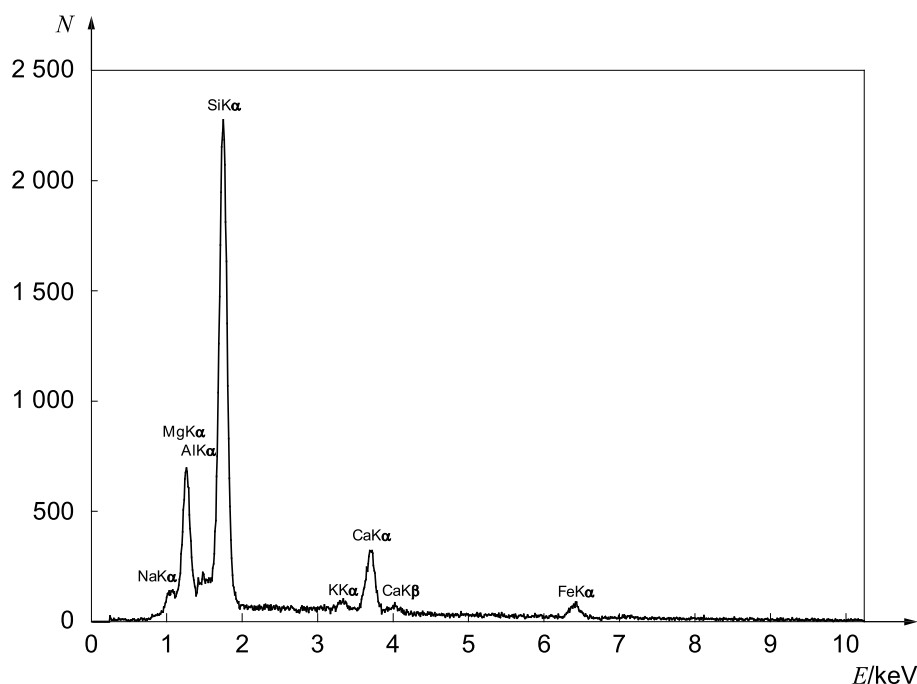
**Figure E.9 — Energy dispersive X-ray spectrum obtained from HSE anthophyllite**

**Key**

N counts

E

X-ray energy

**Figure E.10 — Energy dispersive X-ray spectrum obtained from Bolivian crocidolite****Key**

N counts

E

X-ray energy

**Figure E.11 — Energy dispersive X-ray spectrum obtained from richterite/winchite**

## Annex F (normative)

### Asbestos identification by TEM in commercial materials

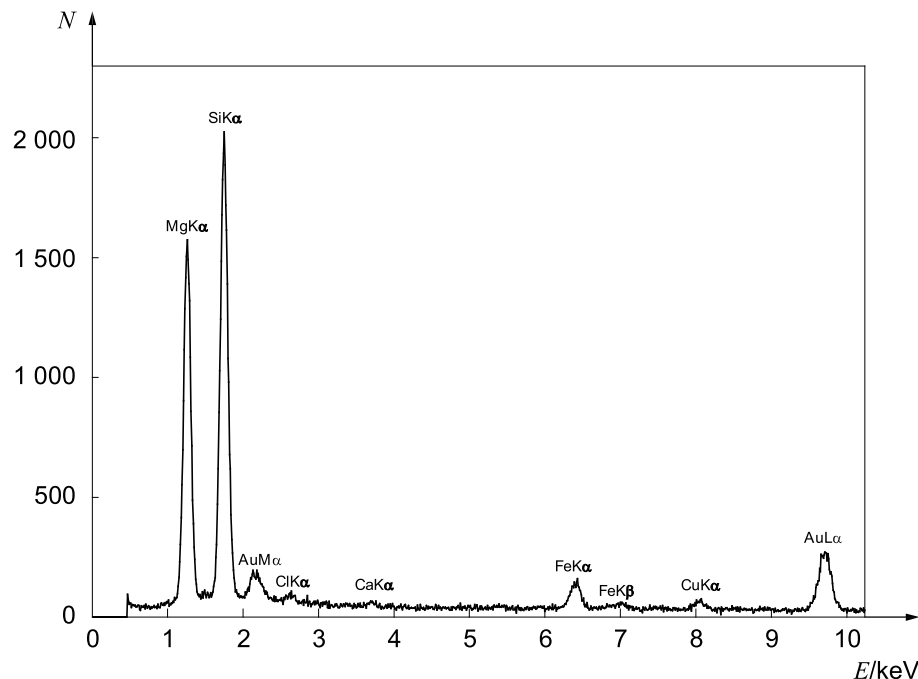
#### F.1 General

For the identification of asbestos in some types of bulk materials, particularly for those in which PLM examination yields ambiguous results, TEM examination can usually resolve the ambiguities and provide definitive identification of the fibres. In most cases, acquisition of an EDXA spectrum provides sufficient evidence to identify any of the asbestos varieties. Discrimination between talc and anthophyllite, however, cannot be reliably achieved on the basis of an EDXA spectrum alone, because the chemical compositions of the two minerals are very similar. Electron diffraction permits discrimination between talc and anthophyllite on the basis of their different crystal structures.

#### F.2 EDXA analysis

Figures F.1 to F.11 are examples of EDXA spectra collected on a TEM operating at 80 kV and using a silicon solid state detector with a beryllium window. The TEM specimens were prepared by the micropipette method from SRM 1866, SRM 1867 and HSE reference asbestos varieties. All specimens were prepared using gold grids in order to avoid interference in detection of the Na  $K_{\alpha}$  peak by the Cu  $L_{\alpha}$  peak which would partially overlap the sodium peak if copper specimen grids were used.

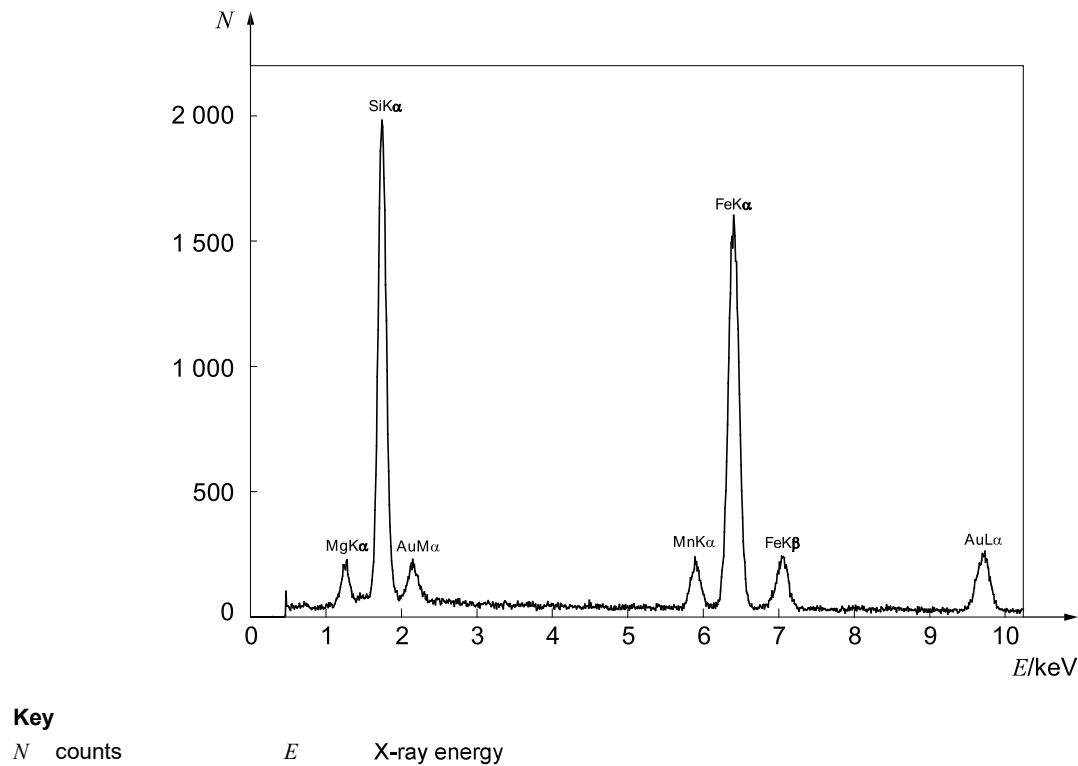
Prior to use of this part of ISO 22262, obtain calibration spectra from the reference standards, using the actual accelerating voltage and the specific X-ray detector.



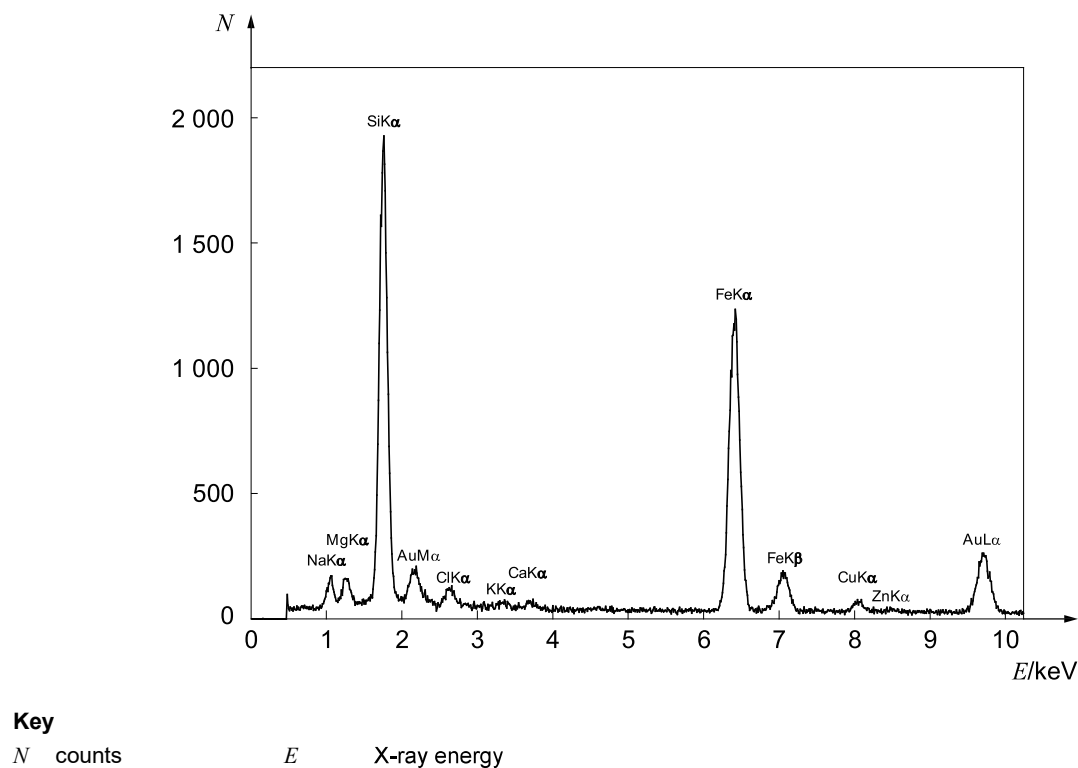
#### Key

$N$  counts       $E$  X-ray energy

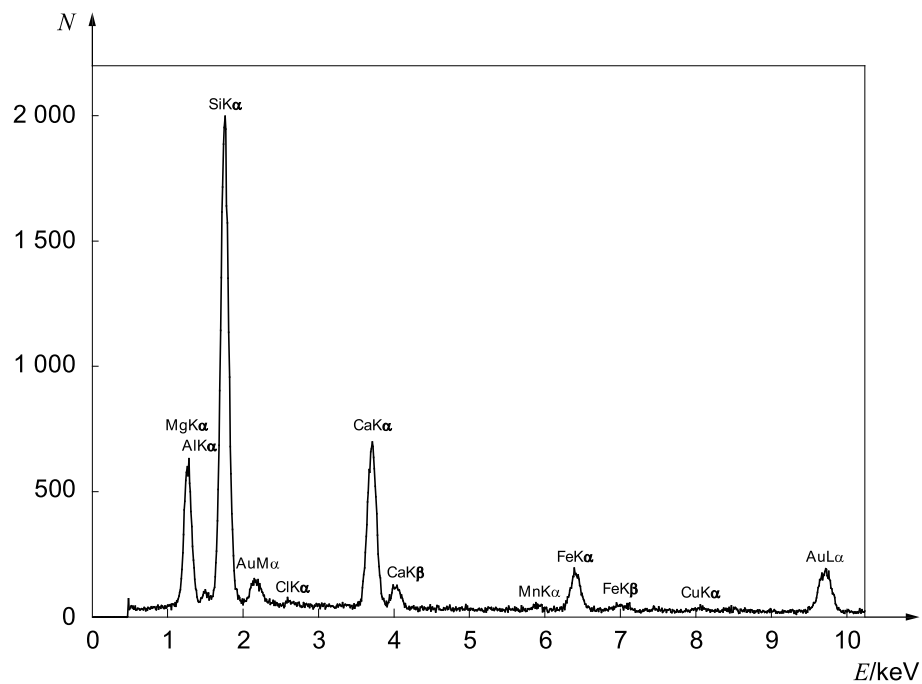
**Figure F.1 — Energy dispersive X-ray spectrum obtained from SRM 1866 chrysotile.  
The gold and small copper peaks originate from the gold specimen grid**



**Figure F.2 — Energy dispersive X-ray spectrum obtained from SRM 1866 amosite.  
The gold peaks originate from the gold specimen grid**



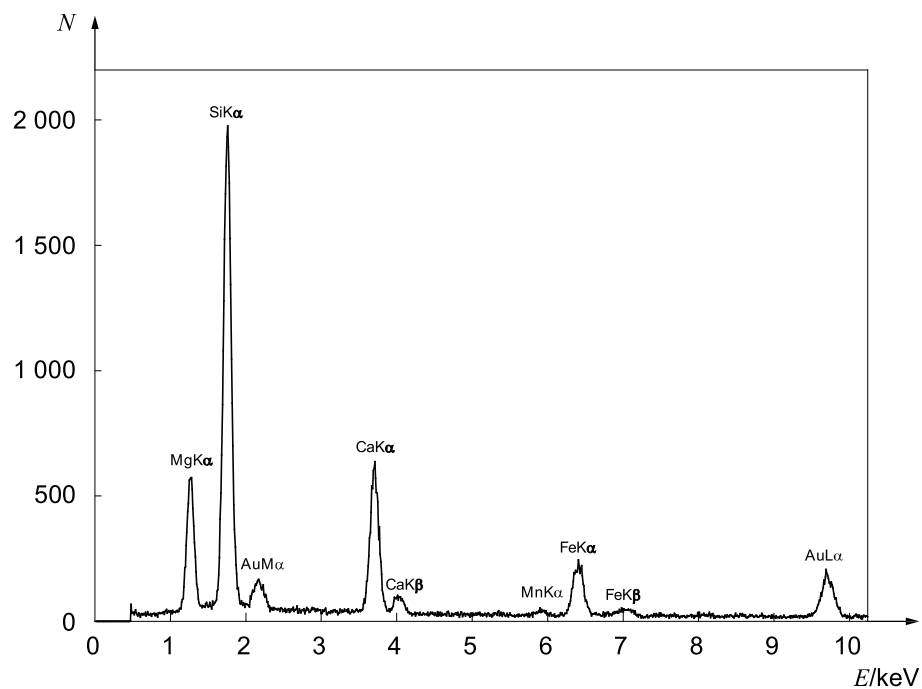
**Figure F.3 — Energy dispersive X-ray spectrum obtained from SRM 1866 crocidolite.  
The gold and small copper peaks originate from the gold specimen grid**



**Key**

$N$  counts       $E$  X-ray energy

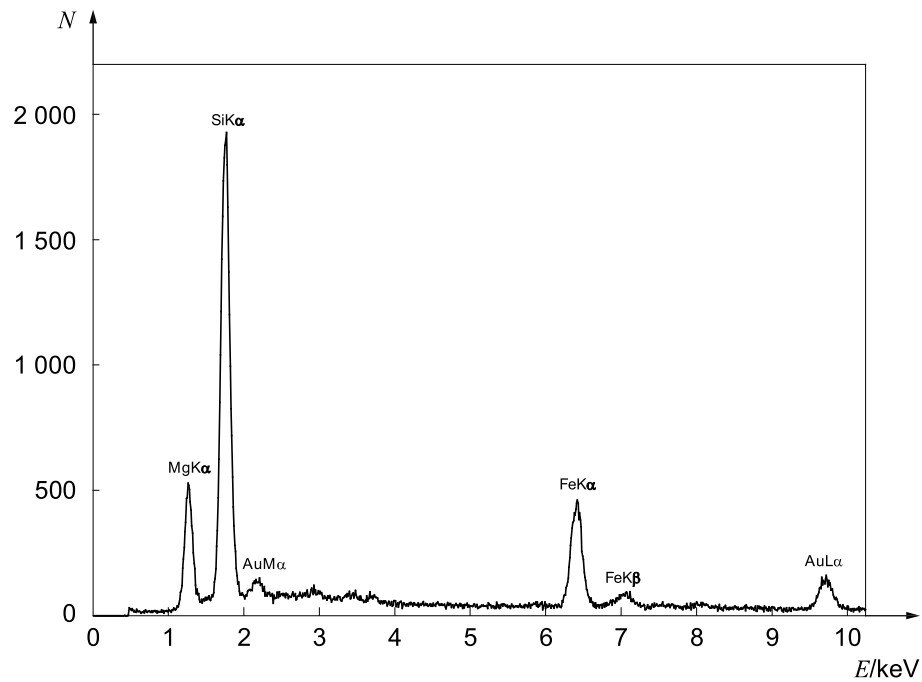
**Figure F.4 — Energy dispersive X-ray spectrum obtained from SRM 1867 tremolite.  
The gold and small copper peaks originate from the gold specimen grid**



**Key**

$N$  counts       $E$  X-ray energy

**Figure F.5 — Energy dispersive X-ray spectrum obtained from SRM 1867 actinolite.  
The gold peaks originate from the gold specimen grid**

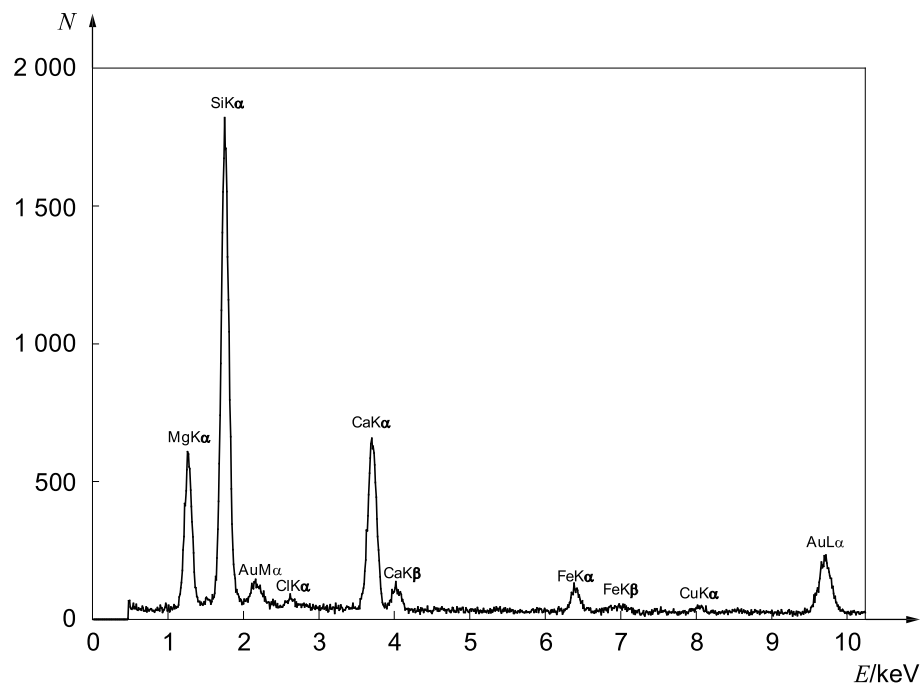
**Key**

N counts

E

X-ray energy

**Figure F.6 — Energy dispersive X-ray spectrum obtained from SRM 1867 anthophyllite.  
The gold peaks originate from the gold specimen grid**

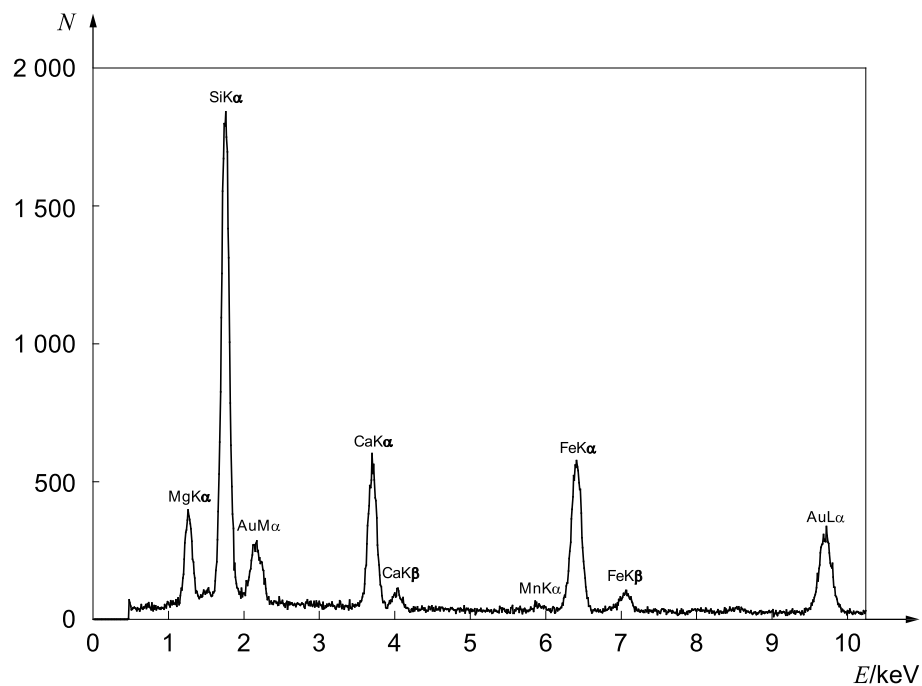
**Key**

N counts

E

X-ray energy

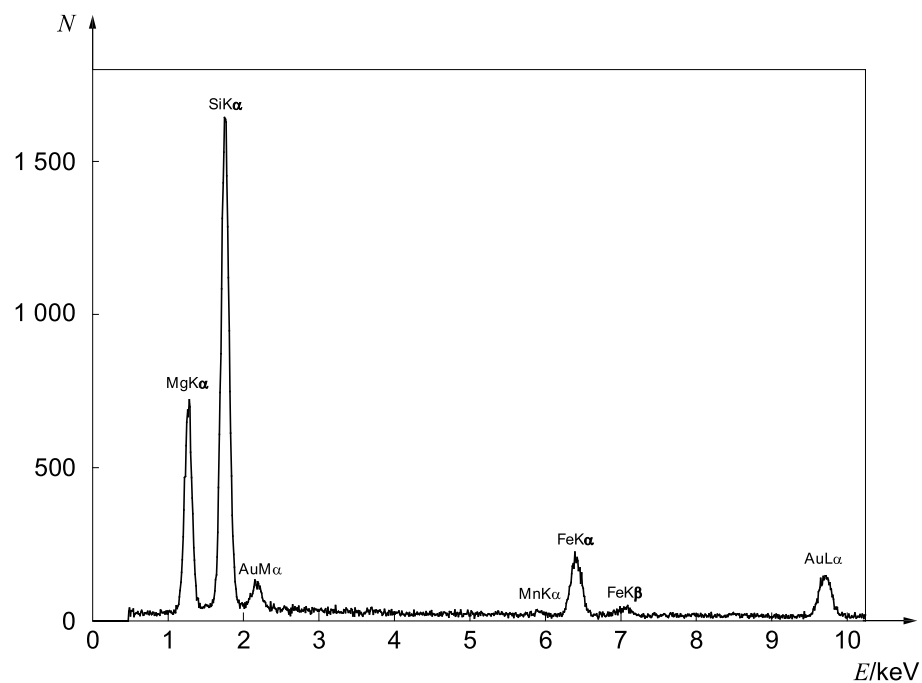
**Figure F.7 — Energy dispersive X-ray spectrum obtained from HSE tremolite.  
The gold and small copper peaks originate from the gold specimen grid**



**Key**

$N$  counts  $E$  X-ray energy

**Figure F.8 — Energy dispersive X-ray spectrum obtained from HSE actinolite.  
 The gold peaks originate from the the gold specimen grid**

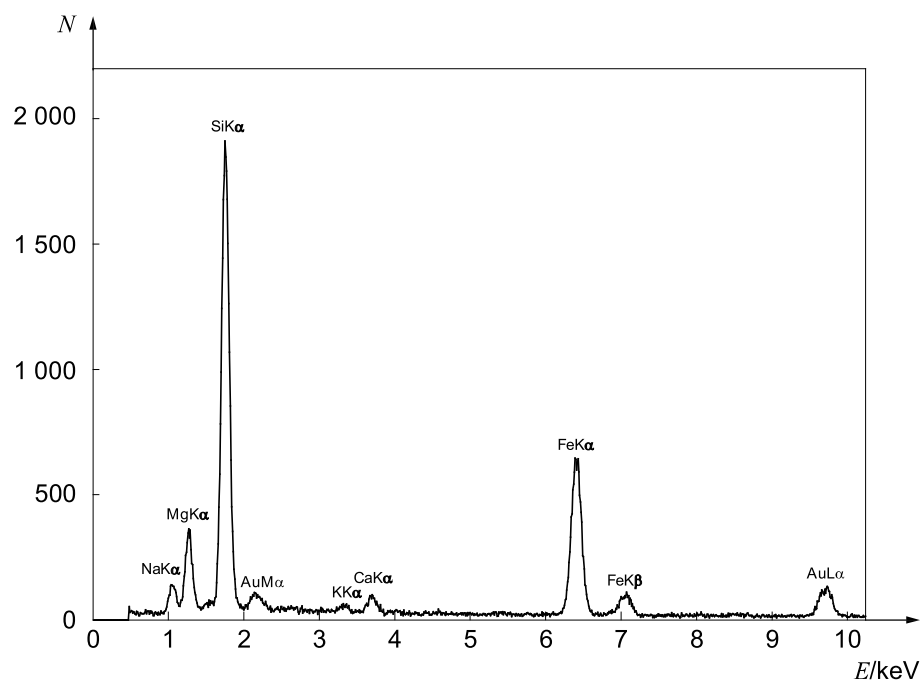


**Key**

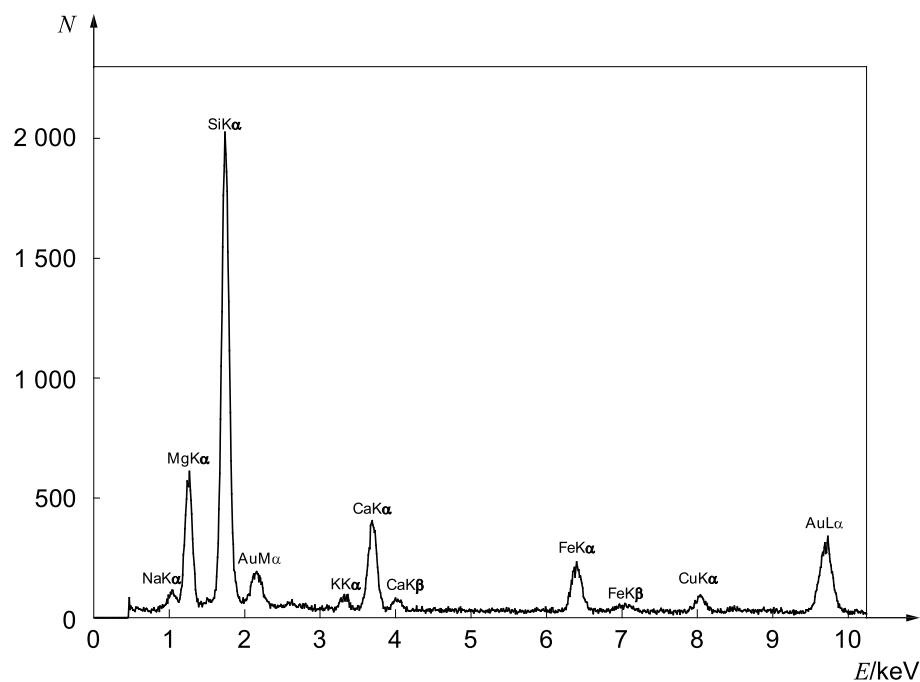
$N$  counts  $E$  X-ray energy

**Figure F.9 — Energy dispersive X-ray spectrum obtained from HSE anthophyllite.  
 The gold peaks originate from the gold specimen grid**



**Key***N* counts*E* X-ray energy

**Figure F.10 — Energy dispersive X-ray spectrum obtained from Bolivian crocidolite.  
The gold peaks originate from the gold specimen grid**

**Key***N* counts*E* X-ray energy

**Figure F.11 — Energy dispersive X-ray spectrum obtained from richterite/winchite asbestos.  
The gold and small copper peaks originate from the gold specimen grid**

### F.3 Electron diffraction

The ED technique can be either qualitative or quantitative. Qualitative ED consists of visual examination, without detailed measurement, of the general characteristics of the ED pattern obtained on the TEM viewing screen from a randomly oriented fibre. ED patterns obtained from fibres with cylindrical symmetry, such as chrysotile, do not change when the fibres are tilted about their axes, and patterns from randomly oriented fibres of these minerals can be interpreted quantitatively. For fibres which do not have cylindrical symmetry, only those ED patterns obtained when the fibre is oriented with a principal crystallographic axis closely parallel to the incident electron-beam direction can be interpreted quantitatively. This type of ED pattern shall be referred to as a zone-axis ED pattern. In order to interpret a zone-axis ED pattern quantitatively, it shall be recorded photographically and its consistency with known mineral structures shall be checked. A computer program may be used to compare measurements of the zone-axis ED pattern with corresponding data calculated from known mineral structures. The zone-axis ED pattern obtained by examination of a fibre in a particular orientation can be insufficiently specific to permit unequivocal identification of the mineral fibre, but it is often possible to tilt the fibre to another angle and to record a different ED pattern corresponding to another zone axis. The angle between the two zone axes can also be checked for consistency with the structure of a suspected mineral.

For visual examination of the ED pattern, the camera length of the TEM should be set to a low value of approximately 250 mm and the ED pattern should then be viewed through the binoculars. This procedure minimizes the possible degradation of the fibre by the electron irradiation. However, the pattern is distorted by the tilt angle of the viewing screen. A camera length of at least 2 m should be used when the ED pattern is recorded, if accurate measurement of the pattern is to be possible. It is necessary that, when obtaining an ED pattern to be evaluated visually or recorded, the sample height shall be properly adjusted to the eucentric point and the image shall be focused in the plane of the selected area aperture. If this is not done, there may be some components of the ED pattern which do not originate from the selected area. In general, it is necessary to use the smallest available ED aperture.

For accurate measurements of the ED pattern, it is recommended that an internal calibration standard be used. Apply a thin coating of gold, or other suitable calibration material, to the underside of the TEM specimen. This coating may be applied either by vacuum evaporation or, more conveniently, by sputtering. The polycrystalline gold film yields diffraction rings on every ED pattern and these rings provide the required calibration information. Alternatively, a calibrated objective aperture can be inserted to determine if the layer-line spacing of the ED pattern is approximately 0,53 nm, as expected for asbestos fibres (Reference [30]). This works well even when viewing a raised screen through binoculars.

To form an ED pattern, move the image of the fibre to the centre of the viewing screen, adjust the height of the specimen to the eucentric position, and insert a suitable selected area aperture into the electron beam so that the fibre, or a portion of it, occupies a large proportion of the illuminated area. The size of the aperture and the portion of the fibre shall be such that particles other than the one to be examined are excluded from the selected area. Observe the ED pattern through the binoculars. During the observation, the objective lens current should be adjusted to the point where the most complete ED pattern is obtained. If an incomplete ED pattern is still obtained, move the particle around within the selected area to attempt to optimize the ED pattern, or to eliminate possible interferences from neighbouring particles.

ED patterns can be particularly useful for differentiating fibrous talc from anthophyllite asbestos, both of which have similar EDXA spectra. ED of talc produces a pseudo-hexagonal pattern that does not change as the fibre is tilted using the goniometer. Anthophyllite asbestos, on the other hand, produces assorted spots appearing and disappearing along layer lines as the fibre is tilted using the goniometer. ED patterns can also be a useful diagnostic tool for chrysotile that is so heavily coated with matrix that EDXA is inconclusive. Detection of the 002, 110, and 130 reflections as shown in Figure F.12 in conjunction with 0,53 nm layer-line spacing confirms the presence of chrysotile.

Analysis of laboratory samples seldom requires zone-axis measurements. However, if a zone-axis ED analysis is to be attempted on the fibre, the sample shall be mounted in the appropriate holder. The most convenient holder allows complete rotation of the specimen grid and tilting of the grid about a single axis. Rotate the sample until the fibre image indicates that the fibre is oriented with its length coincident with the tilt axis of the goniometer, and adjust the sample height until the fibre is at the eucentric position. Tilt the fibre until an ED pattern appears which is a symmetrical, two dimensional array of spots. The recognition of zone-axis alignment conditions requires some experience on the part of the operator. During tilting of the fibre to obtain zone-axis

conditions, the manner in which the intensities of the spots vary should be observed. If weak reflections occur at some points on a matrix of strong reflections, the possibility of twinning or multiple

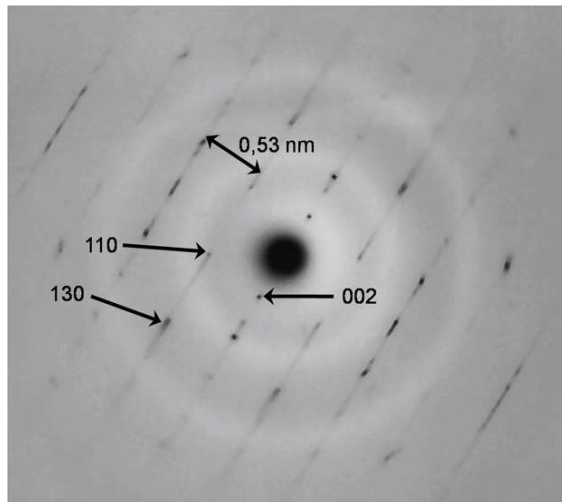


Figure F.12 — Chrysotile SAED pattern

diffraction exists, and some caution should be exercised in the selection of diffraction spots for measurement and interpretation. A full discussion of electron diffraction and multiple diffraction can be found in References [26]–[29].

It is important to recognize that not all zone-axis patterns that can be obtained are definitive. Only those patterns with closely spaced reflections corresponding to low indices in at least one direction should be recorded. Patterns in which all  $d$ -spacings are less than about 0,3 nm are not definitive. A useful guideline is that the lowest angle reflections should be within the radius of the smallest ring of the gold diffraction pattern (111), and that patterns with smaller distances between reflections are usually the most definitive. It is particularly important to recognize that when ED is used to discriminate between different minerals of similar compositions, demonstration that an ED pattern is consistent with the crystal structure of a particular mineral is not proof of identity, unless the ED pattern has also been shown to be *inconsistent* with the crystal structures of the other possible minerals.

Computer programs such as XIDENT (Reference [31]) provide a convenient way to test the consistency of any given ED pattern with the crystallographic data for individual minerals. The XIDENT program is advantageous in that no knowledge of crystal orientation is required; all possible ED patterns at all orientations are calculated and compared with the observed ED pattern. If the results obtained from one ED pattern do not resolve any ambiguity in identification of a fibre, a second ED pattern obtained at a different orientation of the fibre can be examined, and the observed tilt angle between the two orientations can be compared with the theoretical angle calculated from the suspected crystal structure. In order to use the XIDENT program, five spots, closest to the centre spot, along two intersecting lines of the zone-axis pattern are selected for measurement, as illustrated in Figure F.13. The distances of these spots from the centre spot and the four angles shown provide the required data for analysis. Since the centre spot is usually very over-exposed, it does not provide a well-defined origin for these measurements. The required distances are best obtained by measuring between pairs of spots symmetrically disposed about the centre spot, preferably separated by several repeat distances.

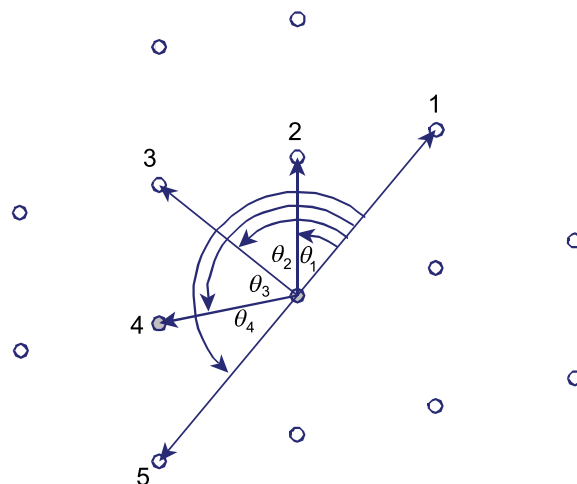


Figure F.13 — Measurement of spacings and angles in a zone axis ED pattern

## Annex G

(informative)

### Example of sampling record

Date:	Samples taken by:
Building and location:	

Room:		Sample identification:
Sampling location:		
Reference:	Plan No:	Position in plan:
Sketch No:		Photo No:
Sample details:		
Comments:		

## Annex H (informative)

### Example of test report

#### Analysis of bulk materials for asbestos by ISO 22262-1

Date of analysis:			
Analyst:		Signature:	
<p>NOTE ISO 22262-1 refers to qualitative analysis of commercial products for asbestos.</p> <p>In this method, polarized light microscopy with dispersion staining is the default procedure for identification of asbestos. If the sample characteristics required the use of either of the optional electron microscope methods to identify asbestos, the method used is indicated. If accurate quantification of asbestos mass fraction in the range below approximately 5 % mass fraction is required for the purpose of determining the regulatory status of an asbestos-containing material, use the appropriate other parts of ISO 22262.</p>			

Sample	Asbestos	Estimated asbestos mass fraction	Non-asbestos fibres	Comments
Sample 20050411-1 Pipe covering Grey corrugated paper	Chrysotile	5 %–50 %	Cellulose Brucite	Sample ashed to remove interfering materials.
Sample 20050412-3 Pipe covering White fibrous material	Amosite Chrysotile	5 %–50 % 0,1 %–5 %	None	
Sample 20050412-4 Fireproofing from beam Blue fibrous material	Crocidolite	50 %–100 %	None	
Sample 20050413-1 Pipe covering Off-white fibrous material	None detected	0 %	Mineral wool	
Sample 20050413-2 Plaster White material	Tremolite	0,1 %–5 %	None	
Sample 20050413-3 Ceiling tile Grey fibrous material	Chrysotile	0,1 %–5 %	Mineral wool Cellulose	Chrysotile too fine to identify by PLM. Chrysotile identified by TEM method.

## Bibliography

- [1] ISO 7348:1992, *Glass containers — Manufacture — Vocabulary*
- [2] ISO 10312, *Ambient air — Determination of asbestos fibres — Direct transfer transmission electron microscopy method*
- [3] ISO 10934-1:2002, *Optics and optical instruments — Vocabulary for microscopy — Part 1: Light microscopy*
- [4] ISO 13794:1999, *Ambient air — Determination of asbestos fibres — Indirect-transfer transmission electron microscopy method*
- [5] ISO 14686:2003, *Hydrometric determinations — Pumping tests for water wells — Considerations and guidelines for design, performance and use*
- [6] ISO 14952-1:2003, *Space systems — Surface cleanliness of fluid systems — Part 1: Vocabulary*
- [7] ISO 14966, *Ambient air — Determination of numerical concentration of inorganic fibrous particles — Scanning electron microscopy method*
- [8] AS 4964:2004, *Method for the qualitative identification of asbestos in bulk samples*
- [9] EN 143, *Respiratory protective devices — Particle filters — Requirements, testing, marking*
- [10] EPA/600/R-93/116:1993, *Test method, method for the determination of asbestos in bulk building materials*. Washington, DC: United States Environmental Protection Agency
- [11] MDHS 77, *Asbestos in bulk materials — Sampling and identification by polarised light microscopy (PLM)*. Sudbury: HSE (UK Health and Safety Executive) Books, 1999
- [12] NF X46-020:2008, *Repérage amiante — Repérage des matériaux et produits contenant de l'amiante dans les immeubles bâtis — Mission et méthodologie* [Location of asbestos — Location of materials and products containing asbestos in buildings — Mission and methodology]
- [13] VDI 3866 Part 1:2000, *Determination of asbestos in technical products — Principle — Sampling and sample preparation*
- [14] VDI 3866 Part 4:2002, *Determination of asbestos in technical products — Phase contrast optical microscopy method*
- [15] VDI 3866 Part 5:2004, *Determination of asbestos in technical products — Scanning electron microscopy method*
- [16] WAHLSTROM, E.E. *Optical crystallography*, 2nd edition. New York, NY: Wiley, 1943
- [17] McCrone, W.C., McCrone, L.B., DELLY, J.G. *Polarized light microscopy*. Chicago, IL: McCrone Research Institute, 1984
- [18] McCrone, W.C. *Asbestos identification*. Chicago, IL: McCrone Research Institute, 1987
- [19] SU, S.-C. Dispersion staining: Principles, Analytical relationships and practical applications to the determination of refractive index. *Microscope* 1998, **46**, pp. 123–146.
- [20] MEEKER, G.P., BERN, A.M., BROWNFIELD, I.K., LOWERS, H.A., SUTLEY, S.J., HOEFEN, T.M., VANCE, J.S. The composition and morphology of amphiboles from the Rainy Creek Complex, Near Libby, Montana. *Am. Mineral.* 2003, **88**, pp. 1955–1969
- [21] EMMONS, R.C. A set of thirty immersion media. *Am. Mineral.* 1929, **14**, pp. 482–483
- [22] TYLEE, B.E., DAVIES, L.S.T., ADDISON, J. Asbestos reference standards — Made available for analysts. *Ann. Occup. Hyg.* 1996, **40**, pp. 711–714

- [23] LEAKE, B.E. Nomenclature of amphiboles. *Am. Mineral.* 1978, **63**, pp. 1023–1052
- [24] LEAKE, B.E., WOOLLEY, A.R., ARPS, C.E.S., BIRCH, W.D., GILBERT, M.C., GRICE, J.D., et al. Nomenclature of amphiboles: Report of the Subcommittee on Amphiboles of the International Mineralogical Association Commission on new minerals and mineral names. *Mineral. Mag.* 1997, **61**, pp. 295–321.
- [25] TIMBRELL, V. Characteristics of the International Union Against Cancer standard reference samples of asbestos. *Proceedings, Pneumoconiosis International Conference*, Johannesburg, 1969
- [26] WENK, H.R., editor. *Electron microscopy in mineralogy*. New York, NY: Springer, 1976
- [27] GARD, J.A., editor. *The electron optical investigation of clays*. London: Mineralogical Society, 1971
- [28] HIRSCH, P.B., HOWIE, A., NICHOLSON, R.B., PASHLEY, D.W., WHELAN, M.J. *Electron microscopy of thin crystals*. London: Butterworths, 1965, pp. 18–23
- [29] ALDERSON, R.H., HALLIDAY, J.S. Electron diffraction. In: HAY, D.H., editor. *Techniques for electron microscopy*, 2nd edition. Oxford: Blackwell Scientific, 1965, pp. 478–524.
- [30] WEBBER, J.S. A simple technique for measuring asbestos layer-line spacings during TEM analysis. *Microscope* 1998, **46**, pp. 197–200.
- [31] RHOADES, B.L. XIDENT — A computer technique for the direct indexing of electron diffraction spot patterns. Dept. of Mechanical Engineering, Univ. of Canterbury, Christchurch, New Zealand, 1976. (Research Report 70/76.)





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